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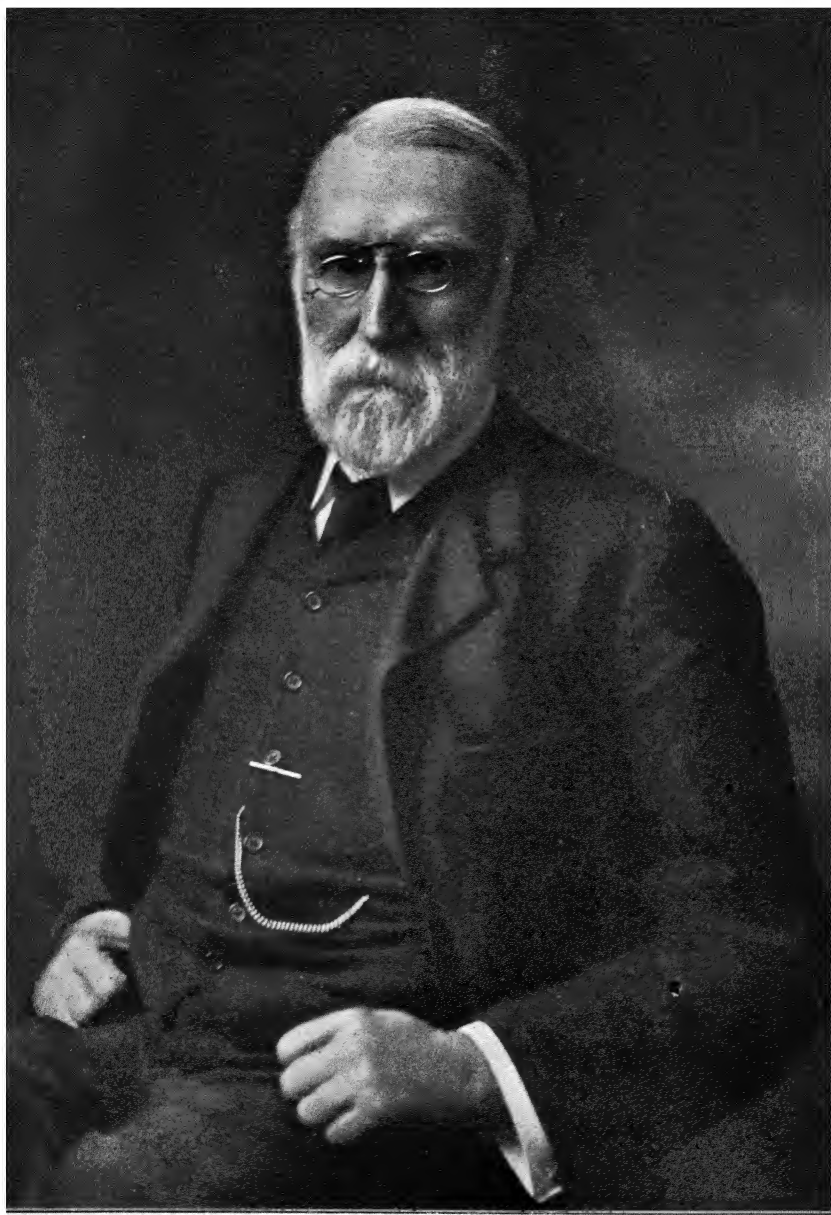












DR. JAMES DOUGLAS.

# TRANSACTIONS

OF THE

## AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS

(INCORPORATED)

WITH WHICH IS CONSOLIDATED THE  
AMERICAN INSTITUTE OF METALS

### VOL. LX

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CONTAINING THE NON-FERROUS AND INDUSTRIAL MANAGEMENT PAPERS  
AND DISCUSSIONS PRESENTED AT THE COLORADO MEETING,  
SEPTEMBER, 1918, AT THE MILWAUKEE MEETING,  
OCTOBER, 1918, AND AT THE NEW YORK  
MEETING, FEBRUARY, 1919.

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NEW YORK, N. Y.  
PUBLISHED BY THE INSTITUTE  
AT THE OFFICE OF THE SECRETARY  
29 WEST 39TH STREET  
1919

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THE MAPLE PRESS YORK PA

## PREFACE

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This volume contains the papers and discussions on non-ferrous metallurgy industrial management and kindred subjects that were presented at the Colorado and Milwaukee meetings in the Fall of 1918, and at the New York meeting in February, 1919.

These papers were printed in *Bulletins* 135 to 146 inclusive, but Vol. LX does not completely supersede any *Bulletin*.

Reports of the Proceedings of the Colorado and Milwaukee meetings are also printed in this volume.



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# PROCEEDINGS OF THE ONE HUNDRED SEVENTEENTH MEETING OF THE INSTITUTE, COLORADO

September 1 to 6, 1918

## COMMITTEE IN CHARGE

Spencer Penrose, *Chairman*

George M. Taylor, *Vice-Chairman*

A. E. Carlton, *Chairman Finance Committee*

J. Dawson Hawkins, *Secretary*

### DENVER

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Richard A. Parker  
T. B. Burbridge

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Frank Bulkley  
Geo. E. Collins

#### Entertainment

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F. E. Shepard  
Howland Bancroft  
B. P. Morse  
J. G. Perry

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#### Finance

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Spencer Penrose  
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A. L. Blomfield, *Treasurer*

#### Registration

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Horace F. Lunt  
J. M. Tippet

#### Arrangement and Entertainment

George M. Taylor  
J. D. Hawkins  
A. E. Carlton  
G. H. Clevenger

A. L. Blomfield  
Thomas B. Crowe  
E. P. Arthur  
Etienne A. Ritter

### CRIPPLE CREEK

Thomas B. Crowe  
E. P. Arthur, Jr.  
N. S. Greensfelder  
A. E. Carlton  
B. F. Hill  
F. G. Willis  
Norris E. Eads  
W. E. Ryan  
Fred Jones  
F. D. Smale  
J. H. Haynes  
L. W. Lennox  
M. R. Valentine

### Ladies

Mrs. T. B. Stearns, *Chairman*  
Mrs. G. H. Clevenger

Mrs. C. Loughridge  
Mrs. H. Bancroft  
Mrs. G. Bancroft  
Mrs. W. D. Leonard  
Mrs. C. Chase  
Mrs. F. H. Bostwick  
Mrs. F. Bulkley

### Ladies

Mrs. G. M. Taylor, *Chairman*  
Mrs. G. H. Clevenger

Mrs. S. Penrose  
Mrs. J. D. Hawkins  
Mrs. L. C. Lennox  
Mrs. H. F. Lunt  
Mrs. E. A. Ritter  
Mrs. L. S. Harner  
Miss F. Dickerman  
Mrs. C. L. Tutt  
Mrs. F. D. Pastorius

### PUEBLO

J. F. Welborn  
H. B. Carpenter  
L. B. Eames  
F. Robinson

It was peculiarly fitting that the American mining engineer, holding a war-time conference, should have chosen for the scene of the meeting the State of Colorado, better endowed, perhaps, than any other State to fill the need for war minerals. The 117th meeting of the American Institute of Mining Engineers reflected in both technical and social features an enthusiastic effort to support every aim of a war-winning character.

Although avoiding any lavishness of display or extravagant entertainment, inappropriate to the times, the Colorado members of the Institute provided a week of well varied instruction and recreation such as can only be offered amid the snow-peaked mountains of the State. The fact that during all but one day of the week "nature's dewy tear drops" fell intermittently failed to detract from the successful execution of the Committee's plans.

Two days in Denver and four in Colorado Springs, from which excursions were made to the Cripple Creek district and Pueblo, comprised the program. On Sunday, Sept. 1, the visiting members and their guests arrived in Denver. Registration at the headquarters in the Brown

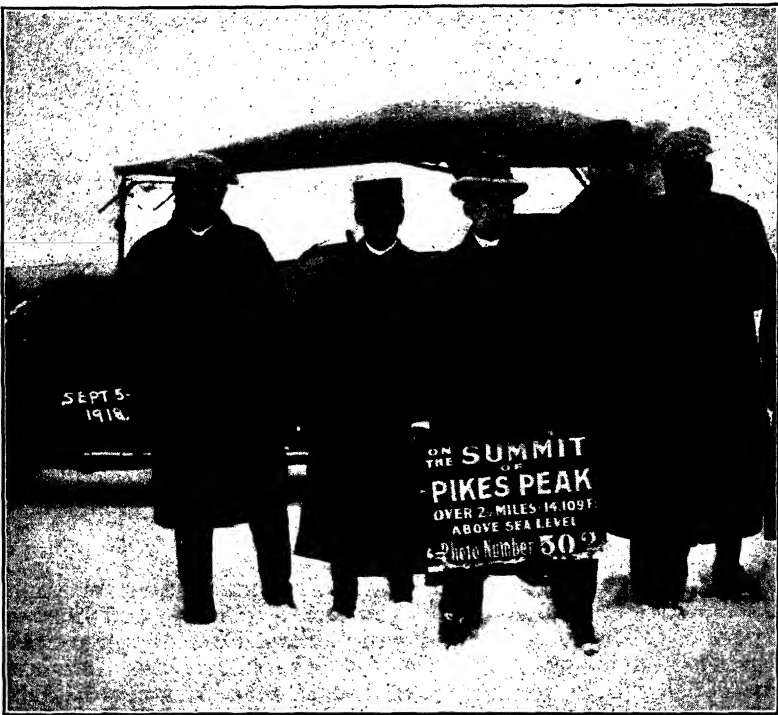


Palace Hotel showed a total of 275 on Sunday night, which number was increased to more than 500 before the close of the meeting.

Sunday was spent in a visit to the City Park Museum in Denver; attendance at a special recital on the municipal organ, said to be the largest and most complete orchestral organ in the world; a special evening display of the electric fountain at City Park, and automobile processions through the City Parks and Boulevards.

On Monday, Sept. 2, shortly after nine o'clock, the first technical session of the meeting was held. Thomas B. Stearns presided and directed the discussion on Metallurgy.

Two hours later, the delegates, conveyed by automobiles, were inspecting the electric ferromanganese furnaces of the Iron Mountain Alloy Co., at Utah Junction, three miles from Denver. This plant, it



was said, is being used experimentally for war purposes preparatory to larger developments. It is in many ways similar to the type of plant at Anaconda, though lacking some of the modern improvements.

Monday noon a luncheon was served to the members, and about one hundred ladies who were guests, in the Denver Club. Following the luncheon, Thomas B. Stearns, Chairman of the Denver Finance Committee of the convention, welcomed the party to Denver and made a bit of history by the remark that inclement weather had come "for the first time in 365 days." Mr. Stearns introduced Mayor W. F. R. Mills, who explained some of the scenic features of Denver and then welcomed the delegates with the words "the City is yours." President Sidney

J. Jennings of the Institute thanked Mayor Mills and the Denver members on behalf of the guests.

Immediately after luncheon the party left in automobiles for Lookout Mountain and Genesee Park, a unique municipal development in that it lies 20 miles from the administrative center. En route, many of the guests were taken to the Herold Pottery Works, at Golden, for an inspection of the process of manufacturing high-grade chemical porcelain.

Following the Lariat trail on Lookout Mountain, over roads built by the City of Denver, the party reached the summit in about one hour and a half. Here is the grave of Col. William F. Cody (Buffalo Bill) covered by native rocks and stones. Many of the party added their pieces to the monument, rough hewn as the man himself, by placing a stone on his resting place.

In the course of the afternoon's tour, which included a trip through Genesee Park and along the banks of the beautiful Bear Creek, a thunder storm broke around the mountains, the greater part being in the clouds beneath the summit of Lookout. It was possible to look down upon the storm from the high altitude and after a few minutes to observe the sun shining through the great white clouds as they parted.

On Monday evening a dinner was served informally in the Denver Country Club to some 300 persons. Following dinner, F. B. Burbridge of Denver established a record as toastmaster by limiting six speakers to a total of 45 minutes. The speakers were Sidney J. Jennings, E. P. Mathewson, H. Foster Bain, C. W. Goodale, Thomas B. Stearns, and Horace V. Winchell.

On the following morning the party journeyed by motor and train to Colorado Springs, arriving at the meeting headquarters, the Broadmoor Hotel, at about noon. At one o'clock a splendid luncheon was served in the hotel dining rooms.

An hour later a memorial service for Dr. James Douglas, in the theatre of the hotel, was attended by every member. President Sidney J. Jennings presided and, after paying a tribute to Dr. Douglas, introduced E. P. Mathewson, representing the Canadian Mining Institute, who, as a fellow Canadian, told of Dr. Douglas' early life in the Dominion; Walter Renton Ingalls, editor of the *Engineering and Mining Journal*, representing the Mining and Metallurgical Society of America, who spoke of Dr. Douglas as a scientist; and T. H. O'Brien, representing the Phelps-Dodge Corporation. The words of these men made a deep impression upon those present. (A full report of this service is given on pages xv to xxi.)

A series of motion pictures, showing the adaptation to industry of soldiers crippled or disabled in war, were later shown in the theatre. These pictures were made by the Canadian Government and loaned to the Institute by the U. S. Department of the Interior. The opportunities thus far opened to cripples from the war, as pictured, surprised many and, as indicated by informal remarks, strengthened their desire to provide "a better place for the cripple than he had held before and to give him the preference."

The remainder of Tuesday afternoon was given over to two simultaneous technical sessions, one on Ore-dressing and Cyanidation, presided over by G. H. Clevenger, and the other on Coal and Coke, at which A. E. Carlton presided. At this latter session many members of the Rocky Mountain Coal Mining Institute were present, though not holding

a joint meeting with the Institute, as planned, because of the absence of their president.

The Ladies' Committee of Colorado Springs furnished an interesting program during the time of the technical sessions, which included visits by automobile to the Garden of the Gods and Glen Eyrie, and tea in the Castle of the Glen. Throughout the week the Ladies' Committee provided many features of special interest to the visiting ladies.

During Tuesday evening a formal reception and dance was held in the Broadmoor Hotel, continuing until after midnight.

Despite a heavy mist on Wednesday morning, Sept. 4, a train of mining engineers was ready for a trip to the Cripple Creek District, at 8 o'clock. A special train on the Colorado Midland Railroad was provided by the hosts, and about 11 o'clock the party was skirting the mountainside in sight of the District. The little town of Altman, said to be the highest incorporated town in the world, was passed and a little later the train reached Victor, where a luncheon was served in a great tent, pitched near the tracks, by the Portland Gold Mining Co. The party then spent some two hours inspecting the Independence mill. During the visit some of the guests were taken through the Cresson mine, producing the highest-grade ore of the district, and others walked or motored down the deserted streets of the town. The romances, the tragedies and the disappointments of gold mining in Cripple Creek were all in evidence, but only as memories. The business of gold mining no longer reads like a story book in Cripple Creek.

The special train returned to Colorado Springs at 6 o'clock. That evening, Dr. Richard B. Moore opened a technical session on Geology and Mining in the theatre of the Broadmoor Hotel, with a series of experiments with radium, presented in a semi-popular manner, and indicating the uses to which radium has been put in the war. This was followed by the discussion of other subjects related to the topic of the evening, H. Foster Bain presiding. During this session, the Secretary presented the following resolution, recommended by the Board of Directors, which was unanimously passed by the members:

**RESOLVED:** That the following minute be entered in the Proceedings of this meeting, and that a copy thereof, signed by the President and Secretary, be sent to the family of Dr. Douglas.

Through the death of James Douglas, this Institute, in common with the professions of mining and metallurgy, and the representatives of liberal learning, technical education, wise philanthropy, and social progress throughout the world, is called to deplore the loss of an inspiring leader, tireless laborer, loyal and helpful friend.

Dr. Douglas' sympathies, quick toward every worthy cause, were especially drawn toward the Institute, because its chief purpose, namely, the free interchange of professional knowledge and experience, commanded his life-long allegiance, not only as a dictate of wise policy, but also as the result of an irresistible generous impulse. He gave freely; he gave "himself with his gift;" and his reward was known of all, even before the record of it, in the gratitude and grief of innumerable friends, was signed and sealed by his death. To them he was not only great, but dear.

Although greeted by a drizzling rain on the morning of Thursday, Sept. 5, and the prediction that snow was falling on the mountain, some 150 of the delegates set out early to make the ascent of Pikes Peak by automobile. The first motor car reached the top of the Peak through a heavy snow fall in about four hours, and before the last car of the party had turned back there was five inches of snow on the ground. Lunch was

served at Glen Cove on the Pikes Peak Highway and, despite the obstacles of the trip, those courageous enough to take it were delighted with the novelty of the adventure. During the afternoon, those who had remained at headquarters and some of those returning from the Peak, visited the Golden Cycle mill on the outskirts of Colorado Springs. Here they observed the precipitation of gold on zinc and the cyanidation of low-grade ores on a vast scale.

Thursday evening was perhaps the busiest single period of the week. Five meetings were held between 8 o'clock and midnight, all of the greatest interest. The first meeting featured a series of motion pictures showing mining and milling methods, welfare work, and the patriotic impulse in the daily routine of the Inspiration Consolidated Copper Co., at Inspiration, Arizona. Dr. L. D. Ricketts supplemented the title explanations of the "movies" with concise facts that made the exhibition almost as instructive as a visit. The pictures were models in the field of cinematography, even though made, in some instances, under handicaps.

President Jennings, at 9 o'clock, presided at a meeting in memory of the 15 members of the Institute known to have made the supreme sacrifice in the war. A service flag of the Institute, showing 845 in service, hung from the stage, and as Secretary Stoughton read the records of those who have died or been killed in the service of the Allies, their pictures were thrown upon the screen. Everyone attending at the Convention joined in paying tribute at this service. (The biographical notices read by the Secretary will be printed in Vol. LXI.)

During the remainder of the evening, three technical sessions were held simultaneously, the principal one being on Petroleum, presided over by R. D. George. The others were a continuation of the discussion of electrostatic precipitation, opened at Monday morning's meeting, and a session on coal-mining problems of today.

Friday morning dawned auspiciously for the trip to Pueblo, and an elaborate program of entertainment for the ladies. The majestic hills forming a background for the hotel were bathed in sunlight and the air was mild and balmy. A special train on the Colorado & Southern Railroad left the Springs at 9 o'clock, taking the party south over the plains to Pueblo and thence to Minnequa, where the works of the Colorado Fuel and Iron Co. are situated.

After an hour's visit to the enormous byproduct coke ovens, installed in July, the members and guests were given luncheon in the company clubhouse situated on the edge of a small lake. The desire of most of the party to spend all their time in the works, which have established, with other steel works, such an important place in war-winning fields, shortened the lunch period, and automobiles were pressed into service in scores to return the delegation to the works. During the afternoon everyone was given an opportunity to inspect the Bessemer and open-hearth processes. Some 40 executives of the plant took groups of the engineers on a thorough tour of inspection. The maximum monthly record for these works in production of steel is 55,000 tons. Some 6000 men are employed.

The return to Colorado Springs was made on the special train about 6 o'clock. The advantages of travel by special train, which was achieved so efficiently by the Colorado Committee, relate to more than mere transportation. The opportunity for social intercourse affords a chance to form many new acquaintances, which are renewed year after year

among the profession, and are often cemented into warm friendships.

During Friday, the ladies of the party were driven by automobile to Crystal Park, where a picnic lunch was served, and at 4.30 o'clock Mrs. Spencer Penrose entertained them at a garden party at El Pomar.

To banquet with a State Food Administrator as toastmaster might seem a dull form of amusement, but the banquet on Friday night, in the dining room of the Broadmoor, which marked the close of the 117th meeting, was a lively finale for the week. Twenty-four songs, printed in booklet form and most of them of a patriotic character, kept the spirits of the party high. A quartet led the singing and their efforts were assisted by various amateur song writers among the diners. Not the least capable among these was one who immortalized the Thursday morning push to the top of the Peak by the following verse—sung to the tune of "There's A Long, Long Trail"—

There's a long, long trail awinding  
Up to the top of Pikes Peak,  
Where the sun is always shining  
And the clouds don't leak.  
There's a long, long time of waiting  
Until the lunch box comes through,  
Till all the mining engineers  
Are sitting down to chew.

Thomas B. Stearns made an inimitable toastmaster; he was introduced by A. E. Carlton.

President Sidney J. Jennings, the first speaker, stated that the aim of the Institute now is to win the war, and completely defeat the Hun. Then he told of some of the work being done by members of the Institute in important posts, and closed with a warning to "steel our hearts against the insidious propaganda of the German Government, which is sure to come, and perhaps very soon when they realize the tide has turned against them." He thanked the Institute's hosts for the entertainment which he characterized as delightful in every respect.

Captain Louis Benett, representative of André Tardieu, High Commissioner of France to the United States, electrified the guests by telling them that the present need is not for transportation or food but "to pour as many tons of steel as you have on the heads of your enemy." Tremendous applause greeted his statement that "America is doubtless France's best friend."

Philip N. Moore then spoke of the importance of the mining engineer in every walk of life, and E. P. Mathewson pictured the work of women in Canadian mining centers. He said they do all kinds of work around smelters except heavy lifting, and that when women take their place in American mining it will be found that "they will do the job better than the men ever did."

C. W. Goodale, who first came to Colorado as a mining engineer in 1876, stated the cardinal necessities to the proper administration of efficient mining, as regards the employee.

Secretary Bradley Stoughton told the guests of the auspicious beginning of the Washington, D. C., Section of the Institute. He then urged the greater use of the library in the Engineering Building in New York, although stating that it is now doing work for more mining engineers and metallurgists than for any other of the great societies in the building. He then explained the value of the Index which first appeared in the

September Bulletin; gave new honor to the Woman's Auxiliary for its war work, especially the founding of a dispensary in France, and expressed the great appreciation of the members and friends for the untiring and thorough efforts of the Colorado members in making the meeting highly successful, none, he said, having exceeded it in varied features.

Horace V. Winchell spoke on the Russian situation, and Dr. L. D. Ricketts closed the banquet, telling, in blank verse, the career of the mining engineer.

Dancing followed the banquet and the ballroom was filled till a late hour, as it had been on each of the four evenings spent in the Springs.

On Saturday, Sept. 7, several of the engineers went to the Leadville district to view the production of the many war minerals of that locality, but most of the party started for their homes on Saturday morning.

### TECHNICAL SESSIONS

#### *Session on Metallurgy*

The session on metallurgy was held on Monday morning, Sept. 2, 1918, at the Brown Palace Hotel, Denver, Mr. T. B. Stearns presiding. The following papers were presented:

Electrolytic Zinc. By C. A. Hansen. (Presented by author and discussed by Sidney J. Jennings and the author. Written discussion by J. L. McK. Yardley.)

The Manufacture of Ferro-alloys in the Electric Furnace. By R. M. Keeney. (Presented by the author.)

The Metallography of Tungsten. By Zay Jeffries. (Presented by title. Written discussion by Sir Robert A. Hadfield.)

The Condensation of Zinc from Its Vapor. By C. H. Fulton. (Presented by title and discussed by E. E. Thum.)

Electrostatic Precipitation. By O. H. Eschholz. (Presented by title. Written discussions by Harmon E. F. Fisher and G. B. Rosenblatt.)

Oxygen and Sulfur in the Melting of Copper Cathodes. By S. Skowronski. (Presented by title.)

The Relation of Sulfur to the Overpoling of Copper. By S. Skowronski. (Presented by title. Written discussion by Philip L. Gill.)

The Practice of Antimony Smelting in China. By C. Y. Wang. (Presented by title.)

#### *Session on Coal and Coke*

The session on coal and coke was held on Tuesday afternoon, Sept. 3, Mr. A. E. Carlton presiding. The following papers were presented:

The Byproduct Coke Oven and Its Products. By William Hutton Blauvelt. (Presented by title and discussed by Graham Bright, S. A. Moss, John I. Thompson.)

The Use of Coal in Pulverized Form. By H. R. Collins. (Presented by the author and discussed by E. A. Holbrook, Captain Walter Graham, Bradley Stoughton, Milnor Roberts, Erskine Ramsay, H. N. Eavenson and the author.)

Carbocoal. By C. T. Malcolmsen. (Presented by the author. Written discussions by F. W. Sperr, Jr., N. W. Roberts, J. M. Fitzgerald, W. R. Cox, F. R. Wadleigh, Charles Catlett, C. M. Barnett.)

Development of the Coke Industry in Colorado, Utah and New Mexico. By F. C. Miller. (Presented by the author.)

Price Fixing of Bituminous Coal by the U. S. Fuel Administration. By Cyrus Garnsey, R. V. Norris and J. H. Allport. (Presented by the Secretary.)

Coal Mining in Washington. By F. A. Hill. (Presented by title. Written discussion by Milnor Roberts.)

#### *Session on Ore-dressing and Cyanidation*

The session on ore-dressing and cyanidation was held on Tuesday afternoon, Sept. 3, Mr. G. H. Clevenger presiding. The following papers were presented:

**The Effect of Oxygen upon the Precipitation of Metals from Cyanide Solutions.** By T. B. Crowe. (Presented by the author and discussed by J. V. N. Dorr, A. L. Blomfield, G. T. Hansen, G. M. Taylor, L. H. Dushak.)

**Roasting for Amalgamating and Cyaniding Cripple Creek Sulfotelluride Gold Ores.** By A. L. Blomfield and M. J. Trott. (Presented by A. L. Blomfield and discussed by J. V. N. Dorr and J. M. Tippet.)

**The Tailing Excavator at the Plant of the New Cornelia Copper Co., Ajo, Ariz.** By Frank Moeller. (Presented by the author and discussed by E. P. Matthewson, C. A. Hansen and Frank Moeller.)

**The Elko Prince Mine and Mill.** By J. V. N. Dorr and C. D. Dougan. (Presented by J. V. N. Dorr.)

**Crushing Resistance of Various Ores.** By L. W. Lennox. (Presented by the author and discussed by R. B. T. Kiliani, C. A. Hansen, V. A. Stout, Rudolf Gahl and the author.)

**Hand-sorting of Mill Feed.** By R. S. Handy. (Presented by title. Written discussions by A. Stanley Hill, W. L. Ziegler, L. O. Howard, Clarence A. Wright, D. C. Bard, S. A. Easton, F. A. Thomson, W. H. Linney and the author.)

**The Automatic Separation of Solution from Solids in the Hydro-metallurgical Treatment of Ore Pulps.** By Bernard MacDonald. (Presented by title.)

**Fine-grinding Cyanide Plant of Barnes-King Development Co.** By J. H. McCormick. (Presented by title.)

### *Session on Economic Geology and Mining Practice*

The session on economic geology and mining practice was held on Wednesday evening, Sept. 4, Mr. H. Foster Bain presiding. The following papers were presented:

**Radium.** By R. B. Moore. (Presented by the author and illustrated by experiments. Discussed by Dr. W. A. Schlesinger, H. J. Seaman, S. A. Moss and the author.)

**Molybdenite Operations at Climax, Colorado.** By D. F. Haley. (Presented by the author.)

**Engineering Problems Encountered During Recent Mine Fire at Utah-Apex Mine, Bingham Canyon, Utah.** By V. S. Rood and J. A. Norden. (Presented by V. S. Rood and discussed by George S. Rice and V. S. Rood.)

**The Relation of Sulfides to Water Level in Mexico.** By P. K. Lucke. (Presented by title.)

**The Mechanics of Vein Formation.** By Stephen Taber. (Presented by title.)

**Pyrite Deposits of Leadville, Colorado.** By Howard S. Lee. (Presented by the author.)

**Fireproofing Mine Shafts of the Anaconda Copper Mining Co.** By E. M. Norris. (Presented by title.)

**Air Blasts in the Kolar Gold Field, India.** By E. S. Moore. (Presented by title.)

**Man Power.** By J. Parke Channing. (Presented by title.)

### *Session on Petroleum*

The session on petroleum was held on Thursday evening, Sept. 5, Mr. R. D. George presiding. The following papers were presented:

**Gaging and Storage of Oil in the Mid-Continent Field.** By O. U. Bradley. (Presented by the author.)

**An Interpretation of the So-called Paraffin Dirt of the Gulf Coast Oil Fields.** By A. D. Brokaw. (Presented by title. Written discussions by W. E. Wrather, E. G. Woodruff and Lee Hager.)

**The Theory of the Volcanic Origin of Salt Domes.** By E. L. DeGolyer. (Presented by title. Written discussion by J. A. Udden and E. L. DeGolyer.)

**A Concrete Example of the Use of Well Logs.** By Mowry Bates. (Presented by the author and discussed by C. A. Hammill, Dorsey Hager and the author.)

**Oil in Southern Tamaulipas, Mexico.** By Ezequiel Ordonez. (Presented by title. Written discussion by V. R. Garfias.)

**Geology of the Oil Fields of North Central Texas.** By Dorsey Hager. (Presented by the author and discussed by C. A. Hammill, C. H. Beal, M. I. Goldman, J. S. Lewis, A. C. Dennis and the author. Written discussion by W. E. Pratt.)

Staggering Locations for Oil Wells. By R. H. Johnson. (Presented by title and discussed by J. L. Lewis.)

Losses of Crude Oil in Steel and Earthen Storage. By O. U. Bradley. (Presented by the author.)

The Possible Existence of Deep-seated Oil Deposits on the Gulf Coast. By A. F. Lucas. (Presented by title.)

Lithology of the Berea Sand in Southern Ohio, and Its Effect on Production. By L. S. Panyity. (Presented by title.)

## THE JAMES DOUGLAS MEMORIAL SERVICE

On Tuesday afternoon, Sept. 3, the Institute held a service in commemoration of Dr. James Douglas, who died at New York on June 25, 1918. President Sidney Jennings presided.

PRESIDENT JENNINGS.—We are met here to show our appreciation of the life of a great man, and we shall gain strength to do our own daily tasks from the contemplation of a career strongly founded, continuously built up, having the star of hope as its guiding light.

Dr. Douglas was an engineer, a scientist, a literateur with a charming sense of style, a benefactor with a singularly wide variety of interests, and a man who had acquired wisdom and understanding, which surpass very great riches. As the poet puts it, "When some beloved voice that was to you both sound and sweetness faileth suddenly, and silence against which you dare not cry aches around you like some disease both strong and new, we poor mortals strive to fill that silence with words of sympathy and appreciation." The very limitation of these words shows our need of the light and guidance which we can acquire from the contemplation of the life of Dr. Douglas.

Other speakers will deal with various phases of the life of Dr. Douglas; I can speak from personal knowledge of only one of his many benefactions to the American Institute of Mining Engineers.

In 1905, Mr. Andrew Carnegie gave to the four national engineering societies of America a sum of money sufficient to erect a large and beautiful building in which to house their activities. He wisely coupled with that gift a proviso that the societies should acquire the title to the ground upon which it was to be built. That entailed upon the slender resources of the American Institute of Mining Engineers a very heavy burden. Many attempts were made to lighten it and contributions were made, but still the burden was heavy, and when I was elected to the Board of Directors, it still weighed heavily upon us.

Dr. Douglas, although he had twice filled the office of President of the Institute and had given much of his time and thought as a Director, came to the rescue and undertook to raise the large sum of money that was necessary to free the Institute from debt. In a comparatively short time, in 1914, largely through his own personal contributions and those of members of the firm with which he was associated, this burden was lifted, and the Board of Directors and members of the Institute were able to breathe once more the air of financial freedom.

In addition to the numerous and large gifts that Dr. Douglas has made to the Institute, by the terms of his will the sum of \$100,000 has been given it for the use of its library, and it is hoped that this sum, together with the yearly contributions made by the four national societies, will bring the library service to that degree of perfection which all those who are interested in the library and its work aim to achieve.



I shall now ask Mr. E. P. Mathewson, a Canadian, to tell us of the early days of Dr. Douglas, who was also a Canadian.

E. P. MATHEWSON.—In my early youth I knew of no name in science to compare with that of Dr. Douglas. He was associated many years with the late Dr. T. Sterry Hunt, and Dr. Hunt was the immediate cause of my coming to the United States from Canada and entering on my professional career in this country.

Dr. Douglas was a man of most benevolent disposition, far-seeing in many ways, who, though possessed of much wealth, thought nothing of money; he had not the love of money at all. The only use he had for money was to do good to those who needed it.

Dr. Douglas was particularly thoughtful of his Canadian fellow countrymen and particularly of those who were engaged in scientific pursuits. The educational institutions of Canada were frequently benefited by his benevolence. McGill University was highly favored by Dr. Douglas, after he learned of the financial difficulties of that institution. McGill, not being granted any aid from the state and relying upon private benevolence, had outstripped its income in giving what it could of educational advantages to Canadians, and it became necessary at one time to have a campaign for more funds. In this campaign Dr. Douglas responded nobly and was the means of getting the necessary funds to go on with the good work of that University. The University from which he graduated, Queens University, was also frequently aided by his benefactions. Altogether, the sums given by him during his lifetime to Canadian institutions would be probably up in the millions, but he was so unobtrusive and so retiring in his disposition that he seldom allowed his name to be used in connection with these matters unless it was possible, by using his name, to influence others to similar benevolence. Anywhere in Canada, if you mention the name "Douglas," you will find people who will say at once, "That was a great Canadian, a man we all revered."

(Mr. Mathewson next read the biography of Dr. Douglas printed in the July 6, 1918, issue of the *Engineering and Mining Journal*. As a biography written by Dr. R. W. Raymond is in this volume and an "Appreciation," by Dr. A. R. Ledoux, was published in our *Bulletin* No. 109, it is hardly necessary to reprint here the account read by Mr. Mathewson.—Ed.)

Dr. Douglas had the broadmindedness to introduce the open door into metallurgy. Prior to his advent into the metallurgical field, the non-ferrous metallurgists in this country, in Canada, and practically all over the world, were absolutely oyster-like toward visitors. No one was admitted who did not have a letter of recommendation from one of the Board of Directors, at least. But Dr. Douglas, early in his career in this country, allowed every one to visit the plant and the mines with which he was connected. He welcomed them, and argued that he was getting as much benefit from the visitors as the visitors were getting from him.

The example of Dr. Douglas was followed by many metallurgists in this country, and today we may say that there is hardly a non-ferrous metallurgical establishment in the United States and Canada to which a person who is honestly seeking information cannot obtain access. Of course, during war times a few precautions are taken for fear that information might get to the enemy. This, of itself, is enough to make Dr.

Douglas called a great man, and to let his name go down to posterity as really the father of open-door metallurgy.

PRESIDENT JENNINGS.—I will ask Mr. W. R. Ingalls, Editor of the *Engineering and Mining Journal*, who was thrown in contact with many of Dr. Douglas's scientific activities in the United States, to tell us of his achievements as a scientist.

W. R. INGALLS.—In the death of Dr. Douglas the Institute lost its greatest member, the mining industry lost one of its greatest exponents, and the world lost a philosopher. Fortunate are we all that his works and his inspiration live after him. The results of his material work will doubtless disappear in the course of time, just as did most of the construction work of the Greeks and the Romans, but just as the teachings of their philosophers survive, so will those of Dr. Douglas, and his inspiration will be one of the world's greatest possessions forever.

The appreciation of how great a man he was will be clearer and keener in the future than it is now. No matter how much we may think we understood him, reflection and meditation will surely reveal to us many things about him that not yet do we see.

Dr. Douglas was a very successful man in material things, and it is one of the remarkable features of his career that this kind of success did not begin to accrue until he was nearly 50 years of age. It is even more marvelous to us in his profession that, although he attained a great age, his great accomplishments were achieved during about 33 years, and those the latter years of his life.

He became a captain of industry, which in itself was a distinction for one who was inherently a philosopher, and he acquired great wealth for which he did not care and which he bestowed bounteously upon many worthy causes; but ambition for material power and a sordid interest in acquiring a great fortune were the furthest of anything from his thoughts. His mind and his fiber were different; his habits were simple; his mode of living was most modest. His thoughts were largely of his studies, and those studies were mainly concentrated upon the improvement of human welfare.

I do not remember when I first became acquainted with Dr. Douglas. I knew of him, of course, from my introduction into professional studies. The beautiful metallurgical process devised by him in connection with the redoubtable Sterry Hunt was one of the things that we were given to ponder upon in the class-room.

I think I first met Dr. Douglas about 20 years ago in connection with his very ingenious muffle roasting furnace, but my intimate association with him was during the last 12 years, when there existed the relations which naturally exist between an editor and his most valued contributor, and also the relations that exist between the fellow members of committees engaged in doing public work.

It has not been until his death that I appreciated the demands that I made upon him and the generosity and the alacrity with which he invariably acceded to them. That is simply one of the revelations of the character of this remarkable man that come to us when he is no longer with us.

Considering his multifarious engagements as the head of the great mining, railway, and other industrial enterprises, I am appalled to think that I could ask him, in the interest of the profession and of the public, to put his work aside and do the writing and the speaking that I and

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Considering his multifarious engagements as the head of the great mining, railway, and other industrial enterprises, I am appalled to think that I could ask him, in the interest of the profession and of the public, to put his work aside and do the writing and the speaking that I and

others wanted him to do. For the very reason that he wanted to aid his fellow men, he was so generous. Oftentimes he would suggest to me editorials that should be written and should be published. Many of the most important editorial expressions that we have made to the public during the last 12 years have been the anonymous contributions of Dr. Douglas, besides those to which he so liberally affixed his name. Oftentimes he would say to me that at the metallurgical works of the Copper Queen Company some important investigations were coming to a head, investigations whereof the profession should be fully informed, and that he would direct his metallurgical men to work up papers on those subjects for the benefit of the industries.

Now, to my mind, the thing that above everything else constitutes Dr. Douglas as one of our great men, a man greater than any of us yet appreciate, is just that interest of his in the promotion of human knowledge and the promotion of such knowledge as would better the welfare of the human race and enable men to work more advantageously.

I think perhaps his first declaration of the principle of the open door, of which Mr. Mathewson has spoken so fittingly, is to be found in his presidential address to this body in 1899, and may I read just a few words from that address,<sup>1</sup> which give the essence of his ideas?

The motives influencing the great body of writers who, without any pay, use the technical journals and such media of communication as our *Transactions*, in order to give to the brethren of their craft the results of their dearly earned experience are various and complicated, but in the majority of cases the impulse originates in the desire for reciprocity and in the hope that others will tell what they know in return for what we ourselves communicate and that, therefore, we shall learn at least as much as we can teach.

Dr. Douglas himself practised what he preached. There was never any secret about operations at the Copper Queen or at any of his enterprises. To every visitor and applicant for information, the helping hand was extended. This spirit spread among other managers, and to that spirit more than anything else ought we attribute the high stage of efficiency to which our American mining and metallurgical industries have come.

If we should turn to the other side of the shield, we should find that in Great Britain these industries have been backward for just the opposite reason. A few years ago I asked a distinguished lead smelter of Great Britain to contribute a paper upon the lead smelting industry of his country. He replied that he could better contribute a paper upon the lead smelting industry of America, for, although he had been engaged in a prominent metallurgical center of Great Britain for 30 years in one of the leading smelting works, and although in the same place there were two other smelting works like his own, he had never, during the 30 years, been into either of them, nor had either of those managers been into his works; but since this great war has been in progress, our British friends have learned the lesson that Dr. Douglas first taught in this country. They are profiting by it, they are collaborating, they are throwing open their doors to one another for an exchange of information to such an extent that they are perhaps outdoing us, and will not unlikely compel us to look to our laurels.

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<sup>1</sup> *Trans.* (1899), 29, 648.

And so it is that the spirit of Dr. Douglas is spreading all over the world, not only through our own country and Canada, but also through Australia and Great Britain, as it will also spread through other parts of the world, and it is for that reason that the time is going to come when the entire world will know him for the great philosopher and the great prophet that we already know him to be.

PRESIDENT JENNINGS.—I will now ask Mr. T. H. O'Brien, who has been delegated by the Phelps, Dodge Corporation, as one who was intimately acquainted with the activities of Dr. Douglas, to tell us of his work in Arizona and the Southwest, and in America generally.

T. H. O'BRIEN.—The death of Dr. Douglas closed a long, honorable, and eventful career, filled with accomplishments that would have occupied fully the lives of several men of less marked ability. Seldom has one man combined in the short span of human life such exceptional achievements. Little can be said regarding his knowledge and ability as an engineer and a scientist that has not already been published, and is known to the members of this Institute. His accomplishments were so varied and extensive that nothing short of the story of his life, written by a competent biographer, can do him justice.

I am not here to give a detailed account of his business career, but rather as a friend and an employee who was associated with him for many years, to tell you something about his work in connection with the company of which he was so long the executive head, and to pay him tribute.

It was in 1880, attracted by some specimens of ore sent from a mine in Arizona, that he paid his first visit to that far off land of which the East then knew so little, which had only lately been made accessible by the construction of a trans-continental railway. It was then that he became associated in a business way with the great Southwest, and this provided the opportunity for his exceptional talents in the development of the mining and railroad possibilities of that part of our country and of Northern Mexico. He never lost the interest thus acquired, and became truly western in his views and preferences.

The Copper Queen mining property at Bisbee had lately been opened, and this attracted him. He interested the late William E. Dodge and D. Willis James, who were at that time metal merchants of New York and partners in the firm of Phelps, Dodge & Co. They began systematic development work. After less than four years the ore began to fail and dark days set in for the enterprise. These reverses only stimulated him to greater effort, and he persisted in his belief that further development would culminate in permanent success. The world knows today how well founded were his perseverance and faith in what has now become one of the greatest copper properties in the world, and it is not too much to say that he alone was the moving cause of that success. It is significant, too, that this achievement came to him when he was nearly fifty years of age, at a time when most men have lost the eager faith and assurance of youth. But this determination was characteristic of the man so long as bodily strength was given to him to follow the direction of his ever versatile and orderly mind.

Perhaps the fact that he entered the mining profession at this time in his life accounts for his having been able to avoid the small prejudices prevalent in the early days of mining and smelting. His was a larger viewpoint, and everything was looked at and considered in a bigger, broader way. His ability to see ahead enabled him to make

provision that insured the steady growth of his operations, and it was this gift of broad vision, combined with the conservative judgment of the original members of Phelps, Dodge & Co., that accounts for the steady growth of the enterprise. He was a dreamer of dreams; he saw possibilities where others saw none; but he lived to see his dreams come true.

He and his associates extended their operations to a consolidation with the Atlanta Company, and later to the districts of Morenci and Globe, in Arizona, Nacozari in Mexico, and afterward to Tyrone in New Mexico. The satisfactory development of these mining and smelting operations under Dr. Douglas' guiding hand soon led him to acquire for the company the coal mines and coking plant at Dawson, New Mexico.

The growth of these numerous enterprises emphasized the urgent need for better transportation facilities. The ability of their founder was equal to the necessity, and Dr. Douglas now turned his attention to the construction of the El Paso & Southwestern Railway system, connecting the various mining and smelting plants with the newly acquired coal property. He thus became a builder of railroads and a master of transportation as successful as he had been in exploiting the mining industry of the Southwest.

What the magnitude of these operations has meant to the Southwest, and especially now, during the great world war, can only be appreciated by those who know the extent of the aid they are giving to the country and its allies in providing raw materials so necessary to the successful carrying on of the war. Truly it may be said that this man did not lay down his cares until he had fully done his part toward winning the great war for permanent peace and equal rights for humanity.

During the development of the different Phelps-Dodge mines, mills, and smelters, there was no man in America, or perhaps in the world, who did more than Dr. Douglas to break down the secrecy as to methods that was prevalent years ago in the great industrial enterprises. He believed in frank, reciprocal relations between competitors in business, and in the greater efficiency that would grow from this policy. Those in charge of his industrial plants were instructed to give every facility to those who earnestly sought to learn. His views on this subject were fully justified, and it became a matter of common knowledge in the business world that his enterprises occupied a unique and enviable position among like institutions. We can only conjecture what great influence this sound policy had in the economic development of the country and the world.

His sympathetic, kindly and democratic nature toward all classes of his employees, and toward those with whom he came in contact, endeared him to each and every one, and to them he was a close personal friend who always had their interests at heart. He was lovingly called "The Professor" by the older prospectors and miners, and was a familiar figure in all southwestern mining camps in the early days.

His philanthropies were many, broad, and effective. In this he avoided publicity, and it will never be known to what extent he aided his fellow man. Individuals alone were not his only charity, but he also went to the assistance of many educational institutions and scientific bodies. He ever stood ready to give counsel, and many a rough place he made smooth for a younger or less fortunate fellow.

He was perhaps the most conversant man on a wide number of subjects that one could ever hope to meet—equally at ease with any subject, unusually well informed on all.

He was always ready to recognize and reward merit, and cared nothing for mere place or position. He was splendidly thoughtful of those who worked with him in his great enterprises, from the highest to the lowest, for their comfort and well being, and no one enjoyed greater loyalty and respect from his associates and employees.

It has seldom been given to one man to see the well ordered success of his life work so completely realized, leaving it, as he did, with the knowledge that he had earned and received the genuine love and respect of all who knew him.

To us who worked with him, his life is now a splendid memory which we will carry with us as an inspiration to the end of our days.



## PROCEEDINGS OF THE ONE HUNDRED EIGHTEENTH MEETING OF THE INSTITUTE, MILWAUKEE, WIS.

The 118th meeting of the Institute was held at the Milwaukee Auditorium, on Tuesday, Oct. 8, to Thursday, Oct. 10, inclusive, 1918, under the joint auspices of the Committee on Iron and Steel (Chairman, Prof. J. W. Richards), and the Institute of Metals Division (Chairman, William M. Corse), and simultaneously with sessions of the American Foundrymen's Association and of the American Malleable Castings Association. It was preëminently a war meeting. It was attended by 104 members of the Institute.

The social features, so far as the members of this Institute were concerned, were slight, but generous and appropriate entertainment was offered to the ladies, consisting of automobile sight-seeing trips, reception, concert and dance on Tuesday evening, a theatre party Wednesday evening, and a banquet in the Auditorium Thursday evening, at which the speakers were Charles M. Schwab, Director General of the United States Shipping Board Emergency Fleet Corporation, Major A. Radclyffe Dugmore, and W. H. Blood, Jr., Assistant to the President of the American International Shipbuilding Corporation.

The opening session was a joint meeting with the American Foundrymen's Association and the American Malleable Castings Association, and was presided over by Benjamin D. Fuller, President of the American Foundrymen's Association. At this time Hon. Emanuel L. Phillipp, Governor of Wisconsin, welcomed the visitors to the city. He said: You will find the people of Milwaukee and of this commonwealth hospitable and above all in complete sympathy with you for the splendid work you are doing, and although, as you pass through the great shops of the city some one may answer you in a foreign tongue, we are all Americans.

The uppermost thought in the minds of the American people, wherever they may assemble, is the winning of the great world war. Without that success, there might be no further reason why we should meet: someone else might tell us what we should do. But we have progressed far enough in that struggle to begin to see the end and it is our kind of an end that we are seeing. In the name of humanity, let us hope that that end will come soon; but in the meantime let us stand firmly together, let us keep the wheels turning until the last gun is fired.

Insomuch as there is every reason for believing that the war cannot last very much longer, it is time that we began to think of what is going to happen and what we are going to do when the war is over. I take a rather optimistic view of that time. I appreciate that some economic changes will come to us, but I do not believe that the business of the country is going to be immediately stagnated or that in the immediate future, at least, there is going to be anything like a paralysis of industries. There is so much in waiting that must be done. One of the first demands will be the improvement of our transportation facilities. The great railroads of the country are wearing out, because it is impossible to secure the labor or the material to keep them in proper repair; but we must do more than merely repair them. The demand is going to come for cheaper

transportation. We are urging the young men to go upon the farms. That is right and proper. Agriculture presents the very best field for the young man returning from military service. However, the farmers of this western country are going to demand better prices than they received before the war. One of the things that can be done to help them get better prices is to furnish transportation at a minimum cost. That will necessitate the purchase of millions of tons of steel. Not only must the tracks be rebuilt and the grades cut down, but the equipment of the railroads must be renewed and the balance of the road rebuilt to meet the demands of a really first-class modern railroad. Public building is being delayed until we can better spare the labor and the material than we can now. So, as I look over the needs of the country, I cannot see why there should be any business depression for many years after the war. I do not mean to say that war prices can be maintained; perhaps they ought not to be. Gradually we must get back to the level that normal times can support. However, the prospect for the future is not so gloomy. Europe is going to furnish a market for our agricultural products for some time and will demand our manufactured material. Besides, we are creating a merchant marine, which will open to us the commerce of the world.

You have assembled here for purposes of your own and, as the Governor of Wisconsin, I am glad you came to us. You brought to us your thoughts and new ideas in manufacture, in the particular line in which you are engaged, and we may be able to give you some information which will be for the mutual good. Happen what will now, let us stand together as one great coöperative organization, keep the wheels going, and, as the boys sing, "Keep the Home Fires Burning."

SECRETARY BACKERT introduced the following communication from a former president of the Association:

MAJOR R. A. BULL.—If I occupied with the American Expeditionary Forces a position of exposure to dangers and hardships, or if I performed a relatively important function in the military organization in France, I would hesitate to voice my sentiments, which in either case might be mistaken for self-praise.

Many things must be done by the non-combatant branches of the American army in France, back of the battle lines, in what is called the Service of Supplies. Those who are doing this work make no pretensions to performing the tasks of heroes, and feel the more keenly their great obligations to their comrades at the front, because of their own assignments in the rear. Many of them have seen, as I have, what a wreck of the yet living body can be made by the enemy's bullet, shell, bomb and gas; have witnessed the fortitude of wounded men under intense suffering; have observed the morale of our soldiers detained for treatment in the rear, keenly anxious to return to the trenches to settle the score with Fritz. Seeing all of this, and realizing how effectively he is hitting the Boche line, my respect for the Yankee fighting man, whatever may be his rank, is supreme. Many of the youths who man the guns, who carry the cold steel over the top, who bridge the streams under the enemy's fire, who minister to the wounded where they fall, are your own kinsmen. How proudly you must bear yourselves in the knowledge that those of your own flesh and blood are bearing this burden! And if perchance those whom you love must make the supreme sacrifice, how glorious a heritage their dauntless courage will leave to you!

It is always comforting to know that our own are in good hands. You have been informed through many channels that the American soldier in France is well cared for. I want to add my endorsement. The medical corps is zealous in its care for the sick and wounded, and in sanitary work. The strictest attention is given to drinking water. Troops quartered in barracks are housed with special regard for ventilation and cleanliness. In the camps in France where I have been stationed there are excellent bath houses, better than those at the camp in the states where

I was formerly on duty. The quartermaster corps is rendering very efficient service in procuring and distributing clothing and other supplies. In most localities, and where conditions permit, the army messes have the most wholesome food, in liberal quantities, well prepared. There is no lack of sugar, wheat flour or meat in the American Expeditionary Force, mainly due, as we realize, to the cheerful self-denial of the folks back home. Just as rapidly as our troops arrive do their supplies seem to precede them.

The American Red Cross is surpassing all its magnificent traditions. It is found everywhere in France, seeking to serve, leaving with those who have felt its influence, grateful recollections that will never fade. Its chief function of caring for those selected by fate as the victims of the enemy's instruments of torture and suffering is being performed with the greatest skill and dispatch, in superb defiance of danger to those who minister. The inspiring devotion of its hard-working, consecrated men and women will constitute one of the most glorious memories of this conflict. Linked as its activities are with every patriotic home in America, its appeal to the sentiment of the Yankee in France makes it his ideal of devoted service that never fails.

The needs of the "Armée Américaine" have been thoughtfully considered apart from purely physical comforts. At the convalescent and rest camps every available means is supplied for cheerful, wholesome entertainment and recreation, with splendid effect on the spirit of the men. By long odds the greatest single factor in maintaining, day in and day out, the morale of the American soldier is the Y. M. C. A. There is the atmosphere of a democratic club, the resort of the finest type of man that has been created—the Yankee buck private.

Tribute has been paid to the splendid work of our allies countless times. After four of the most trying years through which any nation could pass, the French maintain their poise and their vigor to a degree that is amazing. Unstinted praise is demanded by such an inspiring demonstration. The British soldier is entitled to our admiration without bounds. He has been a complete failure—as his own press-agent. As a tenacious, courageous bull-dog who quietly fights on until he or his adversary is done for, he merits our highest esteem. John Bull's allies are under an enormous obligation to these reticent chaps who went quickly from the British Isles and Colonies to the rescue of Belgium and France, and who, without any fuss, have been doggedly seeing the thing through. Do not forget the debt of America to the British navy. And remember that the British empire has to date furnished about 8½ millions of her very best men to save democracy.

I can appropriately testify to the earnest appreciation of the men in the American Expeditionary Force for the splendid work being done by the industrial army in the states. We realize that millions of men and women and many children must labor in America that the vast numbers of her sons in Europe may have the means to finish their task quickly. And we regard those who are unceasingly rendering this service at home and who are best qualified for it, as equal in devotion to duty with those who wear the overseas cap. You realize that every moment of time or ounce of energy wasted in the United States increases the casualty lists of our army. Those who are going through Hell for you and me are confidently looking toward America for that supreme manifestation of speed and efficiency of which her people are capable. Being near to but not of these heroes, without credentials from them but voluntarily speaking for them as an individual, I salute you as brother-patriots, whose sole purpose now is the preservation of liberty for our own and future generations.

The following resolution pledging to the Government the united resources of the iron and steel industry was then unanimously passed:

RESOLVED, by the American Foundrymen's Association, the Institute of Metals Division of the American Institute of Mining Engineers, the Iron and Steel Section of the American Institute of Mining Engineers, the American Malleable Castings Association and the foundry equipment manufacturers of the United States in joint meeting assembled, that every resource of these allied metal trades is again pledged to the Government not only in the production of materials for the conduct of the war, but for the accelerated manufacture of these materials to enable the Government to greatly intensify its prosecution of the war and to bring about a speedy and crushing defeat of the enemy that will lead to his abject and unconditional surrender.

The activities of the army ordnance department, especially as applied to foundry matters, were told by C. S. Koch, of the Cannon

Section of the Production Division, Ordnance Department, Washington. Coöperation between the railroad administration and the metal-working industries was then urged in an address of this title by E. D. Brigham, manager iron ore, coal and grain traffic, United States Railroad Administration, Duluth. The modern methods of transferring skill, illustrated by military films, were shown by Major Frank B. Gilbreth, Providence, R. I.

On Wednesday morning, the Institute of Metals Division conducted a symposium on the conservation of tin, while the iron and steel section covered the programs of both the iron and steel and the coal and coke sessions.

On Wednesday afternoon and Thursday morning the Institute of Metals Division conducted the sessions previously announced for these occasions, dealing mainly with the metallurgy of copper, zinc, brass, bronze, and amalgams.

The plants opened to inspection by visitors to Milwaukee included some very large industrial concerns, such as the Allis-Chalmers Manufacturing Co., the Bucyrus Co., the Wisconsin Gun Co., the Worthington Pump and Machinery Corporation, and many important iron and steel foundries, as well as the pulverized-coal boiler plant of the Milwaukee Railroad and Electric Power Company.

#### INSTITUTE OF METALS DIVISION

The first meeting of the Institute of Metals Division was held at the close of the joint session, with Chairman W. M. Corse presiding. In his address, Chairman Corse said:

The most important event of our year is the affiliation of our Institute with the American Institute of Mining Engineers. It gives us the opportunity of meeting twice a year and of associating, in at least one of these meetings, with men representing the produce of the metals that we all use. The opportunity to study the raw material end of our business has not been afforded at our meetings heretofore and should prove of great value to our members. The affiliation with the American Institute of Mining Engineers gives a permanent headquarters in New York city, the use of the large engineering library in the Engineering Societies' Building, and a permanent secretarial and editorial staff. We, on the other hand, must do our part in making the meetings of our division a success, both by writing papers and by participating in the discussions. Our meetings are generally considered to be excellent from a discussion standpoint. Let us maintain this feature in our divisional meetings and interest in them men who are informed on subjects related in any way to the non-ferrous metal industry.

In these war times, it is particularly necessary to prepare for the reconstruction period to follow by perfecting our manufacturing processes and studying the most efficient methods of transacting our particular business. Any society whose aim is educational has a duty to perform in this respect, and as we represent the non-ferrous alloy and metal industries, it is incumbent on us to see that we are informed of the best and latest practice and furnish the medium for its wide dissemination. The need for maximum production is so great at the present time that it is difficult to find time to do research work, but it seems to be very

necessary that we set aside some money and time in order that we may be ready to produce at the lowest cost and in the most efficient manner, when the times become normal. Our efforts in this direction, through our coöperative work with the Bureau of Standards, have been halted during the war, but it is our intention to continue this coöperative work as soon as practicable.

Our Institute has been the means through which much help has been rendered to the Government and our present affiliation puts us in a position to be of maximum help in this respect. Many of our members have rendered splendid service in technical capacities to the United States, for which we are very glad.

It gives me pleasure to see the generous manner in which our men have responded to any calls made on their time and experience. Let us resolve to make our Institute of Metals Division more of a power in the metal world and to carry on our meetings in such a way that the American Institute of Mining Engineers will feel that they have acquired an energetic and useful member in their household. I want to thank the members for the coöperation they have given me during the year and for the splendid response to our new plan of organization. May the Institute of Metals Division of the American Institute of Mining Engineers be a worthy member of the metallurgical family of which we are now a part.

Secretary F. L. Wolf reported that:

The Institute, on July 1, 1918, had an active membership of 337 and an associate membership of 49, making a total of 386. In the active membership are included the corporation members, each corporation having three members.

Beginning July 1, 1918, the American Institute of Metals became the Institute of Metals Division of the American Institute of Mining Engineers. The advantages of this union were explained in the letter sent to the members on April 18. We retain our identity, elect our own officers as heretofore, hold our meetings as before, at the same time and place as that held by the American Foundrymen's Association and, in addition, a meeting in February, which is held at New York with the American Institute of Mining Engineers. By this affiliation, we secure all the advantages that are offered by one of the largest and best known scientific societies. A glance at our program will show that an excellent program has been provided by the Papers Committee of which Dr. Paul D. Merica is chairman.

The receipts and disbursements for the period of July 1, 1917 to Oct. 5, 1918 are as follows:

*Receipts*

Cash on hand July 1, 1917.....	\$ 740.04
Dues.....	3610.50
Volumes.....	783.96
Emblems.....	21.00
Interest.....	2.00
Refund from Rumford Press.....	19.25
Rental of Electros to Metal Industry.....	5.00
A. F. A.....	250.00
Miscellaneous.....	.37
	<hr/>
	\$5432.12

*Disbursements*

Printing including Postage.....	\$3202.98
Postage.....	114.15
Salaries.....	1025.00
Office Supplies.....	19.91
Refunds.....	50.25
Bond.....	2.50
Insurance.....	37.62
Convention.....	236.10
Miscellaneous.....	25.49
Exchange.....	7.85
Cash on hand Oct. 5, 1918.....	710.27
	<hr/>
	\$5432.12

As officers for the ensuing year, the nominating committee, consisting of Gwilliam H. Clamer, chairman, J. L. Jones, and Alfred Frank, recommended the following, for whom the Secretary was instructed to cast the ballot: *Chairman*, W. M. Corse, Ohio Brass Co., Mansfield, O.; *secretary-treasurer*, F. L. Wolf, Ohio Brass Co., Mansfield, O.; *vice-chairmen*, who will also form the executive committee, Wm. B. Price, Scovill Mfg. Co., Waterbury, Conn.; George K. Burgess, Ph. D., Bureau of Standards, Washington, D. C.; Harold J. Roast, James Robertson Co., Ltd., Montreal, Can.; C. H. Bierbaum, Lumen Bearing Co., Buffalo, N. Y.; W. A. Cowan, National Lead Co., Brooklyn, N. Y.; Sir Robert A. Hadfield, 22 Carlton House Terrace, London, Eng.; W. K. Frank, Damascus Bronze Co., Pittsburgh, Pa.; C. H. Mathewson, Ph. D., Sheffield Scientific School, New Haven, Conn.; Zay Jeffries, Ph. D., Aluminum Castings Co., Cleveland, O.; W. H. Bassett, American Brass Co., Waterbury, Conn.

## TECHNICAL SESSIONS

*Institute of Metals Division*

One session of the Institute of Metals Division was held on Tuesday morning, October 8, Mr. W. M. Corse presiding. The following papers were presented:

The Metallography of Tungsten. By Zay Jeffries. (Presented by the author; discussed by Sir Robert Hadfield, J. C. W. Humfrey, P. D. Merica, and the author.)

Notes on Babbitt and Babbitted Bearings. By Jesse L. Jones. (Presented by the author; discussed by G. H. Clamer and the author.)

The second session was held on Wednesday morning, October 9, Mr. W. M. Corse presiding. The following papers were presented:

Constitution of the Tin Bronzes. By S. L. Hoyt. (Presented by P. D. Merica; discussed by C. H. Bierbaum.)

Oxygen and Sulfur in the Melting of Copper Cathodes. By S. Skowronski.

Relation of Sulfur to the Overpoling of Copper. By S. Skowronski.

(Both papers were presented by W. H. Bassett, and discussed by F. Johnson (written), G. H. Clamer.)

Pure Carbon-free Manganese and Manganese Copper. By Arthur Braid. (Presented by the author; discussed by W. H. Bassett, G. H. Clamer.)

The third session, a symposium on the conservation of tin, was held on Wednesday morning, October 9, immediately following the session scheduled above, and was continued on Wednesday afternoon. Mr. G. C. Stone was in the chair. The following papers were presented:

Babbitts and Solder. By G. W. Thompson. (Presented by W. A. Cowan.)  
 Bronze Bearing Metals. By G. H. Clamer. (Presented by the author.)  
 Pennsylvania Railroad Anti-friction and Bell Metals. By F. M. Waring. (Presented by W. M. Corse.)  
 Solder, Its Use and Abuse. By M. L. Lissberger. (Presented by the author.)  
 The Tin-plate Industry. By D. M. Buck. (Presented by the Chairman; discussed by G. H. Clamer, J. W. Richards.)  
 The Aluminum Bronze Industry. By W. M. Corse. (Presented by the author.)  
 Bronzes, Bearing Metals, and Solders. By G. K. Burgess and R. W. Woodward. (Presented by P. D. Merica; discussed by G. H. Clamer, R. T. Roberts.)  
 Cadmium Resources of the United States. By C. L. Siebenthal. (Presented by P. D. Merica; discussed by M. L. Lissberger, C. W. Hill, F. F. Colcord.)

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The fourth session was held on Wednesday afternoon, beginning at the close of the symposium on tin. Mr. W. M. Corse presided. The following papers were presented.

The Volatility of the Constituents of Brass. By John Johnston. (Presented by the author; discussed by J. W. Richards.)  
 The Effect of Impurities on the Hardness of Cast Zinc or Spelter. By G. C. Stone. (Presented by the author.)  
 Dental Amalgams. By A. W. Gray. (Presented by the author, and illustrated by lantern slides.)

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The fifth session was held on Thursday morning, October 10, Mr. G. C. Stone presiding. The following papers were presented:

Electrolytic Zinc. By C. A. Hansen. (Presented by title. Written discussions by J. L. McK. Yardley and the author.)  
 The Condensation of Zinc from its Vapor. By C. H. Fulton. (Presented by C. C. Nitchie; discussed by E. E. Thum.)  
 The Action of Reducing Gases on Copper. By N. B. Pilling. (Presented by the author.)  
 Notes on Non-metallic Inclusions in Bronzes and Brasses. By G. F. Comstock. (Presented by title.)  
 Fusible Plug Manufacture. By G. K. Burgess and L. J. Gurevich. (Presented by title.)  
 Application of the Spectroscope to the Chemical Determination of Lead in Copper. By C. W. Hill and G. P. Luckey. (Presented by C. W. Hill.)  
 Radium. By R. B. Moore. (Presented by title. Written discussion by W. A. Schlesinger.)

### *Iron and Steel Section*

The session of the Iron and Steel Section was held on Wednesday morning, October 9, Dr. J. W. Richards presiding. The following papers were presented:

The Engineering Work of the National Research Council. By H. M. Howe. (Presented by John Johnston.)  
 The Limonite Deposits of Mayaguez, Mesa, Porto Rico. By C. R. Fetteke and Bela Hubbard. (Presented by title.)  
 The Manufacture of Ferro-alloys in the Electric Furnace. By R. M. Keeney. (Presented by title. Written discussion by E. S. Bardwell, H. W. Gillett.)  
 The Manufacture of Silica Brick. By H. LeChatelier and B. Bogitch. (Presented by title.)

Notes on Certain Iron-ore Resources of the World, N. Y. Section Meeting of May 23, 1918. (Presented by title. 'Discussed by J. W. Richards.)

Recent Geologic Development on the Mesabi Iron Range, Minn. Discussion by Anson A. Betts and J. F. Wolff. (Presented by title.)

The Byproduct Coke Oven and its Products. By W. H. Blauvelt. (Presented by title.)

The Use of Coal in Pulverized Form. By H. R. Collins. (Presented by the author; discussed by A. V. Adamson, J. W. Richards, H. H. Stock, R. F. Harrington, T. A. Marsh, and the author.)

Carbocoal. By C. T. Malcolmson. (Presented by N. W. Roberts.)

Low-temperature Distillation of Illinois and Indiana Coals. By G. W. Traer. (Presented by the author.)

Method of Fixing Prices of Bituminous Coal Adopted by the U. S. Fuel Administration. By Cyrus Garnsey, Jr., R. V. Norris, and J. H. Allport. (Presented by title. Written discussion by E. McAuliffe.)





# PAPERS



## Practice of Antimony Smelting in China

BY CHUNG YU WANG, E. M., A. M., HANKOW, CHINA

(Colorado Meeting, September, 1918)

### I. INTRODUCTION

CHINA now leads the world in antimony production, having contributed during recent years something over 60 per cent. of the world's production. The history of the antimony industry of China dates back to 1897 when the Tai Shing Co. was formed, under contract with the Hunan Bureau of Mines, for the smelting of the ore to crude; and in 1908 the Wah Chang Mining & Smelting Co. was formed for the smelting and refining of the ore and crude to regulus. The writer was then asked to take charge of the erection and control of this first plant for regulus production in China and has, since then, been more or less connected with the antimony industry in this country. This paper contains the results of many years' experience in connection with the Wah Chang Mining & Smelting Co., Changsha; the Pao Tai Mining & Smelting Co., Wuchow; the Loong Kee Smelting Works and the To-Cheng Smelting Works, Hankow; as well as information gathered from various other small smelters, scattered in different parts of the Provinces of Hunan, Hupeh, Kwangsi, and Kwangtung.

An idea of the growing importance of regulus production in this country can be gleaned from the fact that, within a period of about 8 years, the export of regulus from Hunan alone jumped from 3 tons in 1908 to about 6000 tons in 1915; that a production of about 23,000 tons of regulus per year could easily be effected from all the smelters, as tabulated below, most of which were erected a year or two ago under the stimulus of the war booms, if the price at Hankow were to stay somewhere about \$900 (Mexican) per ton, half of what it was at its height, thus enabling the utilization of ores from the hinterlands of the provinces; and even under the present ruling of prices and the closing of the less favored smelters, the production of regulus should not be less than 12,000 tons per year.

The distribution of the number of smelters according to provinces is shown in Table 1:

TABLE 1.—*Distribution of Chinese Antimony Smelters*

Provinces	Number of Smelters	Approximate Tons of Regulus per Day	General Remarks
Hunan.....	10	36	The Wah Chang Co. has a capacity of 20 tons per day. The Loong Kee smelting works is the largest.
Hupeh.....	4	18	
Kwangtung.....	4	6	
Kwangsi.....	2	4	The Pao Tai Co. is the largest.
Total.....	20	64	

II. FURNACES AND SMELTING APPLIANCES

*A. Shaft furnaces and condensing chambers for the volatilization process, to roast the low-grade ore or the liquation residue, generally containing 15 to 30 per cent. Sb, and to condense the volatile trioxide.*

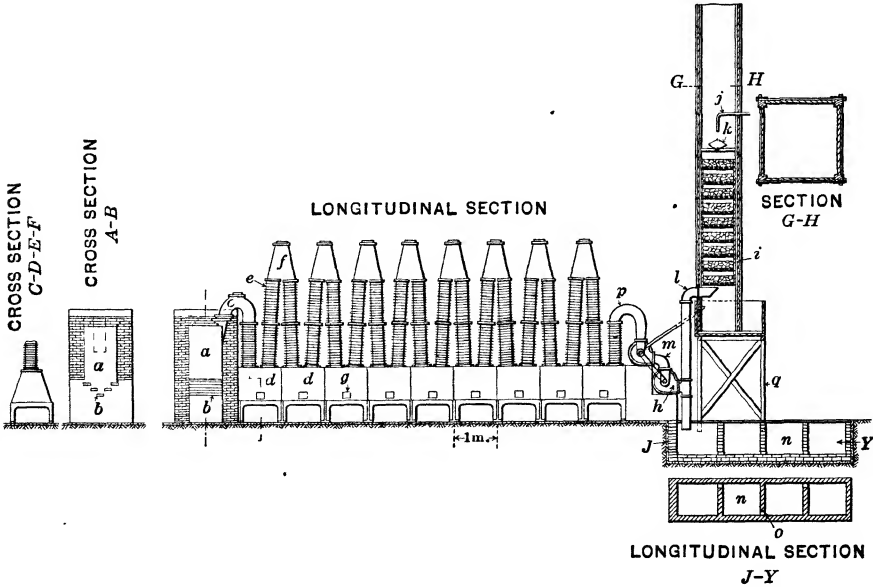


FIG. 1.—SHAFT FURNACES AND CONDENSING CHAMBERS, HERRENSCHMIDT TYPE.

Fig. 1 shows the Herrenschildt type used by the Wah Chang Co. For a full description, refer to the author's book on Antimony.  
Fig. 2 shows the first Herrenschildt type put up at Changsha, 1908.

Fig. 3 is a plan and section of the To-Cheng smelter, designed by the writer. *A*, gas producer. *B*, *C*, reduction furnaces, for reducing the trioxide. *D*, *E*, shaft furnaces, for roasting the low-grade ore or the liquation residue. *F*, *G*, condensing chambers.

Fig. 4 shows the water condensation compartments at the end of the condensation chambers, *F* and *G*, Fig. 3. *a*, suction fans. *b*, basin for collecting the wetted trioxide. *c*, iron pipe for the escape of the sulfurous fume, still holding particles of trioxide. *d*, wooden compartments, arrow showing the direction of flow of the sulfurous fume. *e*, wooden top of the chambers, on which is a constant flow of water, trick-

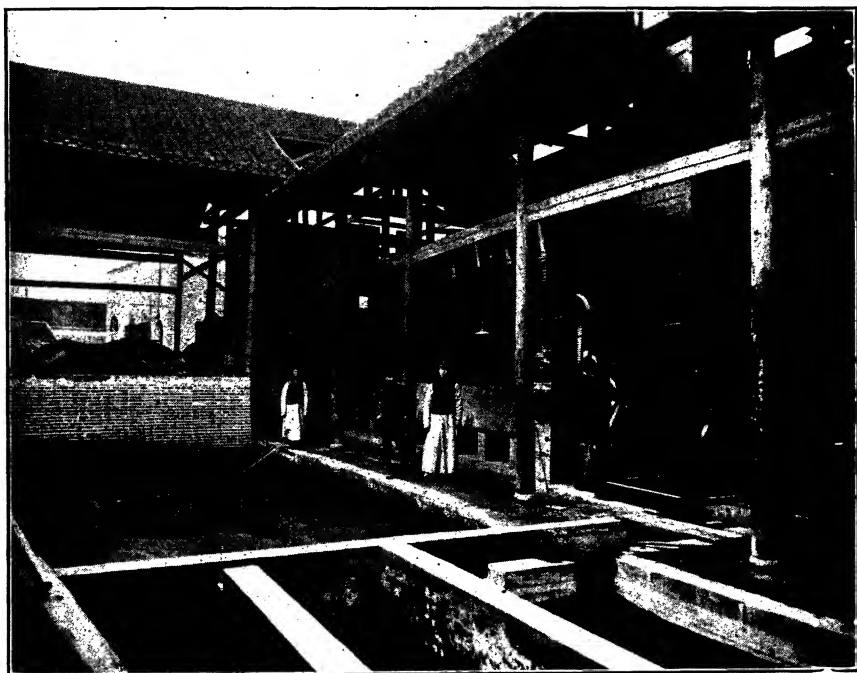


FIG. 2.—HERRENSCHMIDT FURNACES AT CHANGSHA, 1908.

ling down into the chambers below, through a series of holes in the top. *f*, coke. *g*, wooden chimney.

Fig. 5 shows the shaft furnaces and condensation chambers of the Loong Kee Smelting Works.

*B*. Long-bedded reverberatory furnaces for dead-roasting the crude or rich ore to the stable tetroxide.

Fig. 6 shows a general type of furnace adopted by many smelters for this kind of work.

*C*. Reduction furnaces, for the reducing of the trioxide or tetroxide, or for the direct smelting of crude or rich ore, to regulus.



trioxide being condensed in different forms of condensation chambers, as already shown.

*The Wah Chang Mining & Smelting Co.*

(a) *Volatilizing Roasting in a Shaft Furnace.*—The charge consists of a mixture of 100 lb. (45.3 kg.) of ore, ranging from a peanut to a fist in size, and 15 per cent. charcoal. There are approximately two charges per hour

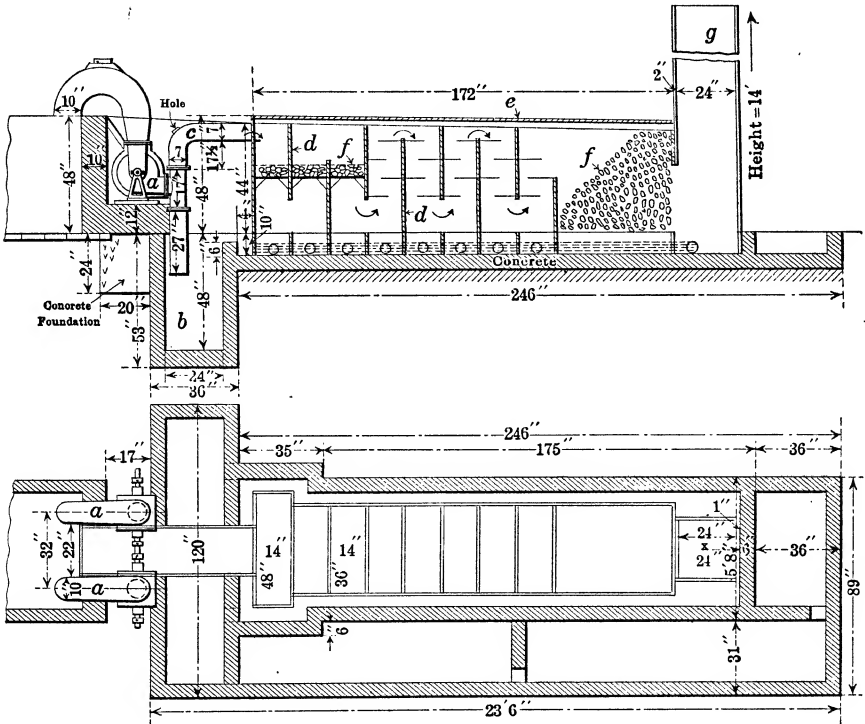


FIG. 4.—WATER-CONDENSATION COMPARTMENTS.

per furnace, depending upon furnace conditions, that is, the interval between charges must be lengthened if it is found that there is fritting of the ores inside the furnace, which must be loosened before another charge is put in. If the charge is composed of liquation residue, the charcoal required for mixing amounts to 20 per cent. for coarse and 24 per cent. for fine. The time interval between charges is about 35 min. for charges of 50 lb. (22.7 kg.) of peanut to fine grain in size; thus the total charge per furnace per 24 hr. amounts to 2240 lb. (1016 kg.). But when the size of the residue ranges from chestnut to fist size, the charges, with the same interval of about 35 min., are then composed of 80 to 90 lb. (36.2 to 40.8 kg.) of residue and 20 to 22 per cent. charcoal, thus mak-



ing a total charge of about 3600 lb. (1633 kg.) per furnace per 24 hr. The scoria discharged is generally found to contain about 3.5 per cent. Sb for ore and about 4.5 per cent. for residue. The average recovery per shaft furnace per 24 hr. from ore of about 25 per cent. Sb, with a total charge of 4480 lb. (2030 kg.), is shown in Table 2:

TABLE 2

	Sb <sub>2</sub> O <sub>3</sub>
From condensation chambers.....	1,000 lb. (453.0 kg.)
From the water-basin (on an average 20 per cent. of the chambers).....	200 lb. ( 90.6 kg.)
From the main flue, 420 ft. (128.8 m.) long (on an average 2 per cent. of the chambers and basins).....	24 lb. ( 10.9 kg.)
Total.....	1,224 lb. (554 5 kg.)

Theoretically the trioxide should contain 83.3 per cent. Sb, but as it is always contaminated with sulfur and other impurities, it contains generally 80.5 per cent. Sb.

Hence the percentage loss in antimony is:

$$100 \times \frac{(0.25 \times 4480) - (1224 \times 0.805)}{(0.25 \times 4480)} = \text{about 12 per cent.}$$

Each furnace requires two workmen per shift of 12 hours.

(b) *Reduction of the Trioxide to Regulus.*—The reduction furnace can smelt two charges per 24 hr. of the following composition:

Sb <sub>2</sub> O <sub>3</sub> .....	3,000 lb. (1,359 kg.)
Charcoal.....	600 lb. ( 282 kg.)
Soda .....	150 lb. (70 5 kg.)

These are thoroughly mixed before being charged into the furnace. The yield of regulus per 24 hr. from 6000 lb. (2718 kg.) of trioxide is from 4100 to 4300 lb. (1858 to 1950 kg.), which is about 70 per cent., exclusive of recoveries from furnace bottoms, flue dust and slag skimming, all of which may amount to at least 5 per cent. of the regulus produced or 3.5 per cent. on the trioxide. Hence the percentage of extraction is 73.5 per cent.; i.e., the loss amounts to 7 per cent., taking the trioxide as containing 80.5 per cent. Sb. Summing up, we have a loss of about 19 per cent. of antimony from ore to regulus. The consumption of coal is 0.55 ton per ton of regulus, and the average life of the hearth bottom is 12 weeks. However, a well constructed bottom may last from 4 to 5 months. Two workmen are required per furnace per shift of 12 hours.

*The Loong Kee Smelting Works*

(a) *Volatilization Process.*—The shaft furnace, as shown in Fig. 5, has a capacity of about 3000 lb. (1360 kg.) of liquation residue per 24 hr. with a consumption of 20 per cent. burnt coal, recovered from the reduction furnace, or 15 per cent. coke. The general practice is to sieve off the fines, to be treated separately from the lumps. Besides the usual condensation chambers at the back of each shaft furnace, there is a main flue of 4517 ft. (1375 m.) in length, to which all the ends of the condensa-

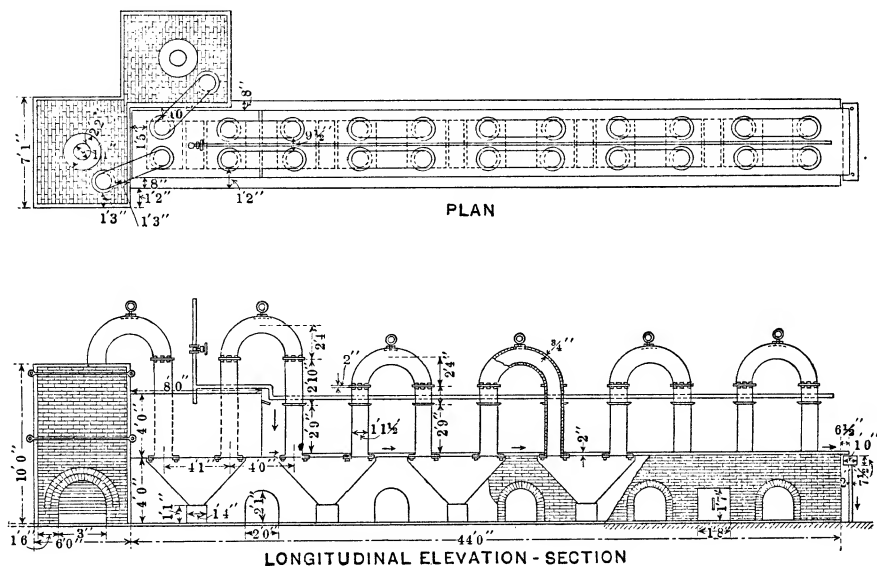


FIG. 5.—SHAFT FURNACE AND CONDENSATION CHAMBERS, LOONG KEE SMELTING WORKS.

tion chambers are joined, leading finally to a series of gunny bags having a total length of 483.75 ft. (147.3 m.). The total wall area of the main flue, which is built of brick, is 104,395 sq. ft. (9702 sq. m.) and that of the bags is 2012 sq. ft. (188 sq. m.). The relative proportion of the amount of trioxide collected from the different parts of the whole system of condensation is as follows: Condensation chambers : Main flue : Bags = 1.65 : 1 : 0.09. As the furnaces treat many grades of liquation residue and ore—the scoria of the residue could be profitably re-treated only when regulus was quoted in New York at \$0.45 (U. S. ) per pound—it is almost impossible to estimate the percentage extraction without having resorted to an elaborate system of sampling and analysis of the charges.

As this has never been done, the following summary of a month's work is given:

	Pound
Liquation residue charged, including scoria resulting there- from and poor ore.....	404,482
Trioxide obtained.....	64,033
Trioxide smelted.....	61,250
Trioxide used for couverture (which is equivalent to 1568 lb. of regulus).....	2,450
Trioxide loss.....	333
Regulus produced.....	39,052.5
Percentage extraction from oxide to regulus, 63.76 per cent.	
Slag skimmings from the reduction furnace.....	5,728
Regulus from re-smelting of the skimmings.....	849
Regulus from smelting the flue dust, generally 8 per cent. of the total regulus produced.....	3,210.4
Trioxide from the water basin.....	1,064
Regulus resulting from the water-basin trioxide.....	228.5

The total regulus amounts to 44,908.4 lb. (20,370.3 kg.). Hence it may be safely assumed that 9 tons of liquation residue are equivalent to 1 ton of regulus. Assuming that the average percentage of antimony in the charges be 17 per cent., the percentage loss from liquation residue to regulus would then be about 30 per cent., which, from my experience, is a close approximation.

Three workmen are required to run two furnaces per 12-hr. shift.

(b) *Reduction of the Trioxide.*—The reduction furnaces take five charges during 24 hr., each charge having the following composition:

Trioxide.....	250.0 lb. (113.3 kg.)
Charcoal.....	37.5 lb. (16.8 kg.)
Soda.....	5 0 lb. (2.6 kg.)

This mixture is divided into two portions, each being covered with 4 lb. (1.8 kg.) of soda after charging. Hence the total amount of soda required for 250 lb. (113.3 kg.) of trioxide, is 13 lb. (5.9 kg.). Sometimes the charge is not divided up; and, in that case, the charge should contain 13 lb. of soda, instead of 5 lb., and should be covered with 30 lb. (13.6 kg.) of skimmings from the previous charge, which has been mixed with 4 per cent. charcoal and 3 per cent. soda. However, often-times the skimmings from the furnaces are smelted separately with the same proportionate amount of soda and charcoal. The amount of coal required to smelt 1 ton of trioxide is about 1 ton. One workman is required per furnace per 12-hr. shift.

*B. Treatment of Crude and Rich Ore.*—Formerly, that is before the present war, most of the crude was exported, but since then, under the stimulus of war conditions, many small plants have sprung up in Hunan to smelt the crude into regulus.

*Wah Chang Mining & Smelting Co.*

The crude is first ground into powder in a Chilean mill and is then fed into a double-deck roasting furnace, which can dead-roast 2500 lb. (1133 kg.) of crude to the stable tetroxide in 24 hr. with two workmen. In order to increase the output, a long-bedded reverberatory furnace was installed, which can dead-roast 2000 lb. (906 kg.) of crude per 24 hr. with a consumption of 700 lb. (317 kg.) of coal. Two workmen are required per furnace per shift of 12 hr. The tetroxide resulting therefrom is mixed with 6 per cent. soda and 20 per cent. charcoal and is charged into the reduction furnace in the same way as in smelting the trioxide. It is claimed that a recovery of 63 to 65 per cent. is possible.

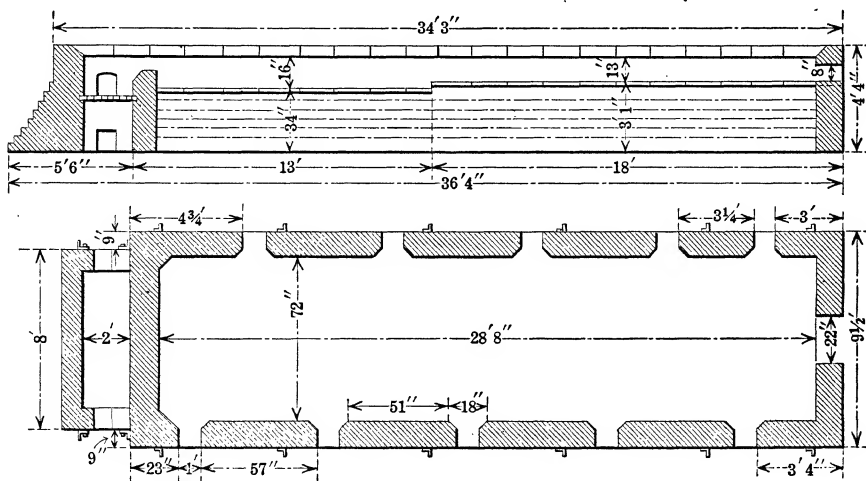
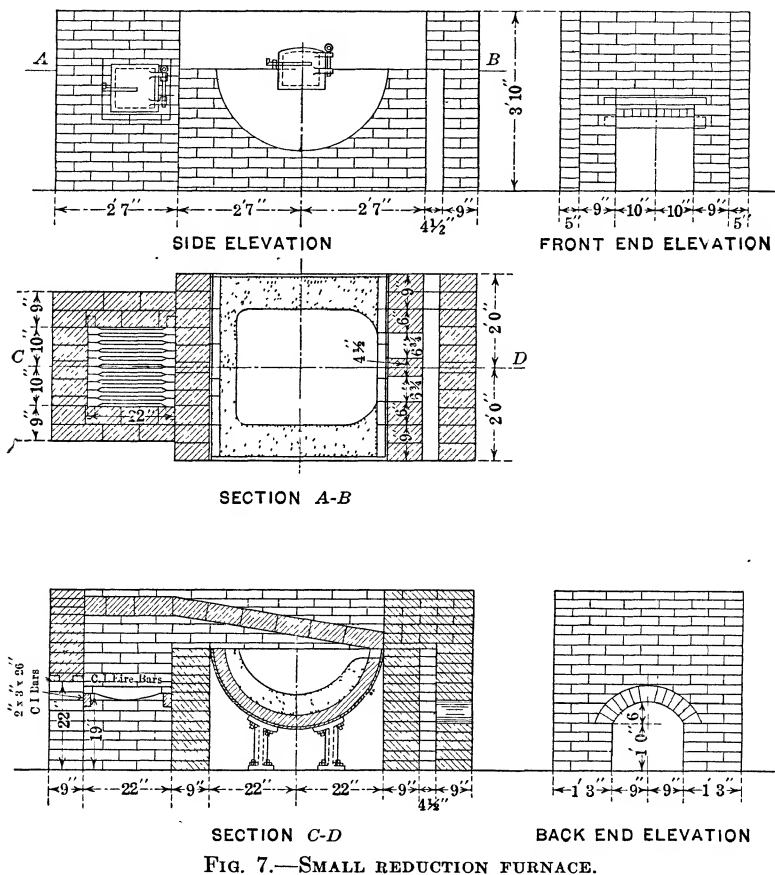


FIG. 6.—REVERBERATORY ROASTING FURNACE.

*The Pao Tai Mining & Smelting Co.*

The reverberatory furnace, shown in Fig. 6, can dead-roast 1975 lb. (895 kg.) of crude per 24 hr., with a consumption of 755 lb. (342 kg.) of coal. Two men are required per furnace per shift of 12 hr. The roasted crude, *i.e.*, the tetroxide, after having been mixed with 15 per cent. charcoal and 5 per cent. soda, is then charged into the reduction furnace shown in Fig. 7, which requires about 1300 lb. (589 kg.) of firewood as fuel and two men to work it, per 24 hr. Each charge contains 300 lb. (136 kg.) of tetroxide and requires 6 to 7 hr. for its smelting. On account of the poor quality of crude used, the average yield from crude to regulus is 57.68 per cent., including recovery from flue-dust and skimming. The treatment of rich ore is exactly the same as that of crude,

except that proportionately more soda and less charcoal are required for mixing and that the fuel consumed per ton treated is correspondingly higher, as more heat is required for the fluxing of the siliceous and argillaceous gangue.



### *The Loong Kee Smelting Works*

This is the only works in China at which crude has been smelted direct to regulus. Each furnace, shown in Fig. 8, can smelt three charges of 200 lb. (90.6 kg.) crude each, per 24 hr. with two workmen. Each charge contains the following ingredients: 200 lb. (90.6 kg.) of crude, mixed with 20 lb. (9.1 kg.) of charcoal, charged first into the furnace, then on top of the charge is spread 100 lb. (45.3 kg.) of soda, and on top of this 88 lb. (39.8 kg.) of iron scrap, preferably sheet scrap, such as waste tinned sheet. The regulus thus obtained has to be re-smelted with a

little crude to take up any iron which may be present, sometimes as much as 9 per cent.

Any grade of ore can be smelted directly in this way, but only with proportionate increase of soda and decrease of tin scrap as poorer ores are smelted; but there must come a limit when it is prohibitive to treat any poorer ore this way on account of the amount of soda required and

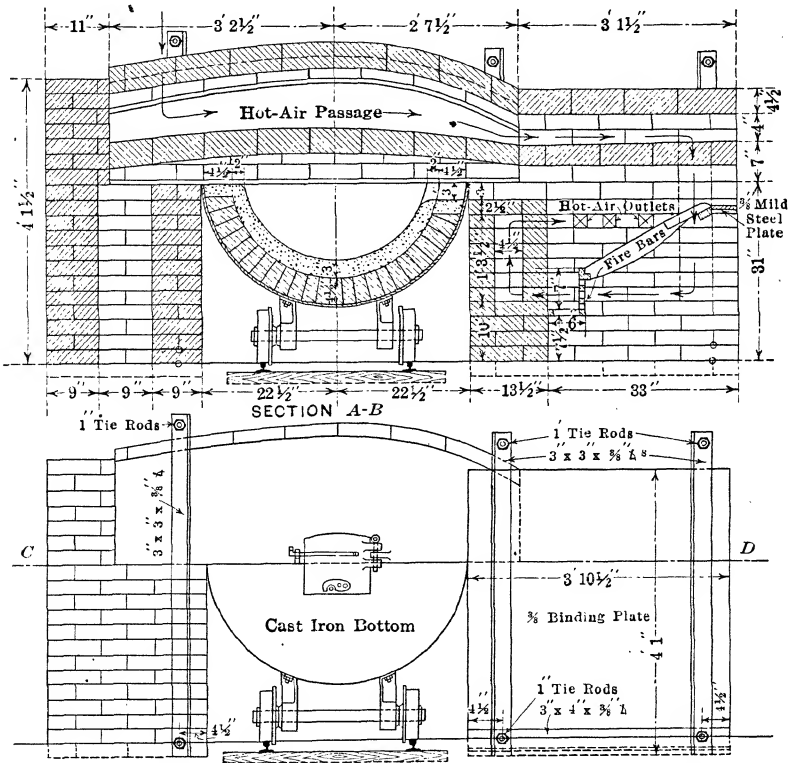


FIG. 8.—SMALL REDUCTION FURNACE WITH REMOVABLE BOTTOM.

of the fuel consumed. An exceptional instance was found in this works in the direct smelting of the rich liquation residue, containing at most from 25 to 30 per cent. Sb, when regulus soared up to \$0.45 (U. S.) per pound in 1915. In this case the charge was as follows:

Liquation residue.....	250 lb.	(113.3 kg.)
Good soft coal.....	22 lb.	(10.0 kg.)
Sheet-iron scrap.....	20 lb.	(9.1 kg.)
Iron sulfide (a byproduct from the previous charges).. <td>10 lb.</td> <td>(4.5 kg.)</td>	10 lb.	(4.5 kg.)
Soda.....	60 lb.	(27.2 kg.)

The time required to smelt this charge is about 9.5 hr., with a production of 46 lb. (20.8 kg.) of regulus.

*C. Treatment of Natural Oxide.*—The natural oxide of antimony is found mostly at one locality in Sin Hua District, Hunan, but smaller quantities are also found in Kwangsi Province. Its supply has been gradually decreasing so that, as a source of antimony ore, it is losing its importance and only a very limited quantity is now smelted by the small smelters at Changsha. The percentage content of antimony in

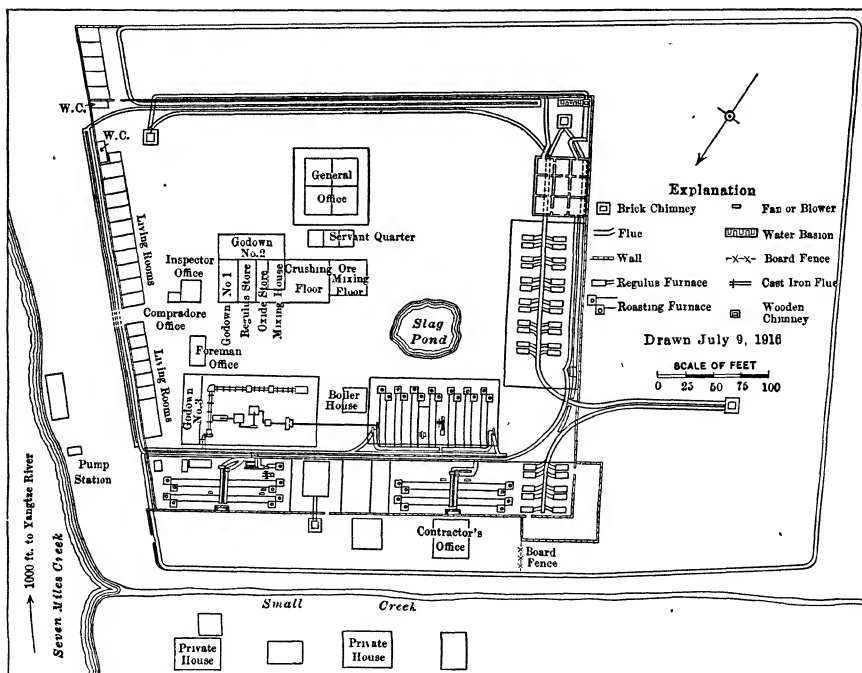


FIG. 9.—PLAN OF THE LOONG KEE SMELTER.

the hand-sorted ore, that can be treated with profit, is from 30 to 50 per cent. Its treatment in the Loong Kee Smelting Works is as follows, the charge having the following composition:

Natural oxide . . . . .	100 lb. (45 3 kg.)
Soda . . . . .	10 lb. (4 5 kg.)
Charcoal . . . . .	13 lb. (5 9 kg.)

Of course, the amount of soda and charcoal required is increased and decreased respectively in proportion to the decreasing antimony content of the ore treated, and *vice versa*. The coal consumption is generally high, being approximately 5 tons to 1 ton of regulus produced for the particular grade of ore, with a percentage extraction of 33 per cent.

In some cases when a small amount of the sulfide is so intimately intercalated with the oxide ore as to render their separation by hand impossible, the following proportions for the charge are adopted:

Natural oxide..	100 lb. (45.3 kg.)
Soft coal.....	9 lb. (4.1 kg.)
Iron sulfide..	5 lb. (2.3 kg.)
Soda..	30 lb. (13.6 kg.)

*D. Discussion of the Use of Couverture.*—As everyone knows, the market always demands good stars with fern-like structure on the slab of regulus before its acceptance. The appearance of such structure does not actually indicate the relative purity of the regulus, but is only the result of cooling it under the cover of a properly prepared starring mixture—in the absence of a proper English term, I shall call it by its French equivalent *couverture*—whose fusion point is lower than that of antimony, which is 630° C. Of course, when the regulus contains impurities like sulfur, arsenic, lead or iron to any appreciable quantity, its surface shows it by specks, by a leaden appearance or by an ill-defined appearance of the fern-like structure. On the other hand, I have seen many a slab of regulus, with impurities above what are considered to be the limits imposed by buyers, whose stars are still bright and well-defined with all the appearance of well refined regulus. Now since the buyer demands such unnecessary adornment on the regulus, he has to pay for it; for the costs for starring 1 ton of regulus amount to from \$10 to \$60 (Mexican), according to the market price of the antimony compound used. I have worked and tried out the different mixtures for preparing the couverture shown in Table 3, which have been adopted, the one or the other, according to local conditions, by various antimony smelters in China:

TABLE 3.—*Couverture Mixtures*

Ingredients		I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII	XIII	XIV
		Pao Tai	Pao Tai	To-cheng	Loong Kee Tai Wo			Pao Tai	Wah Chang	Wah Chang			Loong Kee	Pao Tai	Pao Tai
Smelters using the mixtures.....															
Crude (high-grade)...	Sb <sub>2</sub> S <sub>3</sub>	15	15	2.2	4½	7	3 to 4	5	.....	.....	.....	.....	.....	6	7
Antimony tetroxide (well-roasted crude)	Sb <sub>2</sub> O <sub>4</sub>	24	24	13.2	...	...	...	30	.....	.....	.....	.....	.....	.....	14
Antimony trioxide...	Sb <sub>2</sub> O <sub>3</sub>	...	...	...	20	18	32	.....	85	4 to 5	8	40	7	16*	
Potash.....	K <sub>2</sub> CO <sub>3</sub>	10	11	7.5	.....	.....	.....	.....	.....	1 to 2½	1	1			
Soda ash .....	Na <sub>2</sub> CO <sub>3</sub>	1	3	.	5	4	2 to 4	5 to 6	15	.....	.....	4	1	1	½
Charcoal. ....	C	1¼	1¼	5.7	½	½	1	1	.....	.....	.....	.....	.....	½	½

\* Pure flue dust.



The proper procedure for charging any of the above mixtures is as follows: The compound, after being thoroughly mixed, is immediately charged into the reduction furnace as soon as the skimming is finished. The doors are closed and vigorous firing is maintained; as soon as it is observed that the mixture is completely melted, ladling commences. Each ladle dips into the molten metal and, in coming out, picks up a certain quantity of the molten couverture, which, when poured out together with the metal into a hot mold, completely covers the metal on all sides. Ladling must be done rapidly for each moldful—four or five ladles to one slab of regulus according to the size of the ladle used. The thickness of the solidified couverture varies from 1 to 2 mm. on all sides except the top, which varies from 5 to 7 mm. The amount of couverture required for each charge varies from one-sixth to one-fourth the weight of the regulus produced. It is remarkable that generally the weight of the solidified couverture hammered off from the slab after cooling is less than the original weight of the mixture put in by about one-third, due indubitably in part to volatilization and in part to wall-fluxing during melting. The couverture can be used over again the second or third time, sometimes with an addition of a little soda, until it is so contaminated with impurities that it cannot produce any good stars. Then the wornout couverture is mixed with the ordinary charge of trioxide or tetroxide and is calculated as an equivalent amount of soda required for that charge. In connection with this, the practice at the Loong Kee and the Pao Tai smelters may be mentioned: The Loong Kee smelter uses for every 14 tons of regulus produced 1.14 tons of couverture which, according to No. XII of Table 3, contains 1 ton of trioxide and 0.14 ton of soda. The Pao Tai smelter uses 1 ton of couverture No. XIV for 14.5 tons of regulus.

*E. General Discussion on Smelting.*—Practically all of the shaft furnaces mentioned for the volatilization process are of the same type, except with minor differences in details as to the form of the grate-bottom and as to size. The ideal ore for treatment in such furnaces is one containing antimony from 15 to 25 per cent.; ore above 40 per cent. Sb would partly volatilize and partly liquate, and part of the liquated product would combine with the volatilized trioxide to form an oxysulfide compound, commonly known as antimony glass, which causes fritting at the lower zone of the furnace. Even with ore from 30 to 35 per cent. Sb, some fritting would occur, necessitating the maintenance of the furnace at a higher temperature than necessary for poorer ores. Hence the paradox: the richer the ore, the higher the percentage of coke required for the charge. It is possible to volatilize an ore of about 20 per cent. with 4 to 6 per cent. charcoal, the temperature being thus kept at low red-heat. On the other hand, with liquation residue, which ranges from 15 to 25 per cent., the problem is different. Here

we have a material that loosens up at a very low red-heat, thus partially blocking up the furnace and preventing the free passage of the air upward. To remedy this, some works use a forced draft, while others use an excess of coke, amounting to 30 to 50 per cent. of the charge, thus increasing the porosity of the charge. One can frequently tell when the furnace is blockaded or when the suction fan is choked with the trioxide, especially if water is introduced into it, by observing the appearance of a reddish tint in the issuing trioxide fume, which would be otherwise white with the furnace working under normal conditions. A plausible explanation for the appearance of this reddish tint in the otherwise white trioxide is found in the formation of the compounds  $\text{Sb}_2\text{S}_2\text{O}$ ,  $\text{Sb}_2\text{O}_3 \cdot 2\text{Sb}_2\text{S}_3$ ,  $\text{Sb}_2\text{O}_3 \cdot 2\text{Sb}_2\text{S}_3 \cdot 4\text{H}_2\text{O}$  or  $\text{Sb}_2\text{S}_3 \cdot 2\text{H}_2\text{O}$ . Some sulfide particles, from want of free oxygen, might be volatilized as such at temperatures above  $550^\circ \text{C}$ ., and, taking up the moisture still in the ore, would form a hydrated sulfide, which is red in color. Another fact, which seems to substantiate such an explanation, is that whenever a new furnace, or one which has been standing idle for some time, is started, the trioxide fume is frequently colored red at the commencement and continues so until the furnace is well under way. The explanation here is that there must be present a certain amount of moisture in the furnace, and it is this moisture that is taken up by the volatilized sulfide to form the above-mentioned red hydrated sulfide.

As to the different forms of condensation chambers, not enough data are available to enable one to decide definitely which is best. It is obvious that the elaborate flue and bag systems of the Loong Kee Smelting Works should recover more of the volatile trioxide than others without such equipment.

One must have been struck by the comparative smallness of the reverberatory furnaces adopted for the reduction and smelting of the oxide or crude. Some of the reasons that can be enumerated for such practice are:

1. Facility in the ladling of the metal together with the couverture.
2. Comparatively small output of the smelters.
3. Cheapness of installation and of repairing.
4. The production of better stars on the slab of regulus.

Undoubtedly for such small types of furnace the fuel consumption is high; but it is still questionable whether, in view of the above reasons, one is justified in installing reduction furnaces larger than those of the Wah Chang Works, already described.

Regarding fluxes for smelting the trioxide, tetroxide, or crude, the best flux from standpoint of higher percentage of extraction, lower consumption of coal and of cheapness, is soda ash. That this is so is indicated in Table 4, and has been borne out in my personal experience:

TABLE 4.—*Fluxes for Antimony Smelting*

Flux	Formula	Molecular Weight	Ratio of Equivalent Fluxing Power	Melting Temp., Deg. C*	Price per Ton at Hankow, Pre-war	Actual Price of Equivalent Amount
Soda ash.....	$\text{Na}_2\text{CO}_3$	106	1.00	920	\$84	\$84
Common salt...	$\text{Na}_2\text{Cl}_2$	117	0.91	960	140	154
Potash.....	$\text{K}_2\text{CO}_3$	138	0.77	1,150	117	152
Glauber's salt..	$\text{Na}_2\text{SO}_4$ +10H <sub>2</sub> O	322	0.33	1,280	45	135

\* Taken from *Physico-Chemical Tables*, by J. Castell-Evans.

At one time, toward the end of 1915, when soda was so scarce as to command a price of about \$450 per ton, several trials were made of some cheaper fluxes, such as common salt, potash, and Glauber's salt, but invariably the results obtained were unsatisfactory. Among the many experiments tried, Table 5 illustrates the proportionate amount of the different fluxes used to smelt the trioxide:

TABLE 5.—*Amounts of Flux Required*

	1	2	3	4
Antimony trioxide . . . . .	250.0	250.0	250.0	250.0
Soda.....	.....	6.5		
Common salt . . . . .	8.0	3.0		
Potash... . . . .	9.5	3.0	19.0	
Glauber's salt.. . . .	.....	6.0	.....	37.5
Charcoal.....	37.5	37.5	37.5	37.5

Using charges made up of the sulfide and the oxide, either trioxide or tetroxide, according to the compositions given by Herrenschildt, Pelatan or Basse,<sup>1</sup> I have repeatedly obtained very poor results. We should not be surprised that this is so if we remember that chemically the sulfide and the oxide of antimony do not decompose each other, but combine to form what is known as antimony glass, which is irreducible by means of charcoal.

#### IV. COST OF SMELTING

I shall give two detailed cost sheets, one before the war and one during the war, to show the difference in the cost of smelting as affected by the price of materials.

<sup>1</sup> C. Y. Wang; "Antimony", 107, 120 and 121. London, Griffin, 1909.

*A. The Pao Tai Mining & Smelting Co. (1911).—Cost of materials and labor:*

General labor per shift of 12 hr. . . . .	\$0.233
Furnace men per shift of 12 hr. . . . .	0.25
Coal per ton. . . . .	11.20
Firewood per 100 lb. . . . .	0.30
Charcoal per 100 lb. . . . .	1.00
Soda per 100 lb. . . . .	4.00

TABLE 6.—*Cost of Smelting Per Ton of Regulus*

	From Crude	From Hokong Ore of about 34 Per Cent. Ext.
Crushing crude or ore	\$2 52	\$4 68
Crushing the oxide.	0.39	0 52
General labor. .	0 31	0 14
Labor for roasting furnace. . .	5.87	4.23
Coal for roasting furnace. . .	11.45	10.05
Labor for reduction furnace. .	1.83	3 42
Firewood for reduction furnace.	13.47	26.69
Coal for reduction furnace	0.60	1.30
Labor for mixing charges. . . .	0.36	0.54
Coal in charges . . . . .	2.26	3.24
Soda in charges . . . . .	7.85	17 86
Other miscellaneous labor	.....	0.20
Couverture. . . . .	6 53	10 05
	\$53.44	\$82 92
Add: Cleaning and boxing . . . .	\$5.95	
Iron ware. . . . .	1.64	
Repair to furnaces	1 80	9 39
	\$62 83	\$92 31

*B. The Loong Kee Smelting Works (1915).—Cost of materials and labor:*

Coal per pound. . . . .	\$0.0046
Charcoal per pound. . . . .	0.017
Soda per pound . . . . .	0.03
Coke per pound . . . . .	0.007
Tinned scrap iron per pound . . . .	0.013
Antimony trioxide per pound . . . .	0.25
Labor per shift of 12 hr. . . . .	0.40

TABLE 7.—*Cost of Smelting Per Ton of Regulus*

	Liquation Residue by Volatiliza- tion Process	Direct Smelting of Crude	Natural Oxide about 33 Per Cent. Ext.	Natural Oxide about 14 Per Cent. Ext.	Direct Smelting of Rich Sul- fide Ore 40 Per Cent Ext.
Volatilization process:					
Power plant.....	\$17.70				
Furnace men.....	15.00				
Labor for spalling and mixing charges.....	7.06				
Miscellaneous labor for carrying charges and discharging oxide	2 81				
Coke.....	31.25				
Reduction furnace:					
Soda, charcoal, etc., for charge.....	17.00	\$80.00	\$43.00	\$139.20	\$100.00
Coal for reduction furnace.....	21.00	25.00	54.00	200.00	45.00
Furnace men.....	2.60	6.60	8.00	15.00	6.00
Other miscellaneous labor.....	0.15	0.20	0.50	1.50	0.40
Crushing oxide.....			9.00	21.00	
Boxes, labor for cleaning regulus and for mixing charges for re- duction furnace.....	\$8 60				
Couverture..... 60 00	68 60	68.60	68.60	68.60	68.60
	\$183.17	\$180.40	\$183.10	\$445.30	\$220.00

## V. SOME SUGGESTIONS FOR FUTURE IMPROVEMENT

Due to the lack of capital, and of mutual coördination and coöperation among the smelters and the short-sighted policy of the directors of the different companies, there has not been any marked progress in the metal-lurgy of antimony in China. Obviously an important line of future progress should be in the direction of eliminating the heavy losses due to volatilization of the metal during the successive steps of treatment. To be conservative, let us assume the total loss to be 20 per cent.; then for an annual production of, say, 20,000 tons of regulus, the loss would amount to 4000 tons of regulus, which, at the present market price at Hankow of \$300 per ton, would give an appalling dead loss of \$1,200,000 to the whole industry. I do not believe that we can totally eliminate this loss, but I do believe that, with the adoption of some such proposals as outlined below, the loss could be kept down to 10 per cent., or possibly less, thus effecting an annual saving of at least \$600,000, which would more than pay back the capital outlay for such alterations and additions to the existing plants.

My proposals are:

A. Adoption of a mechanical roaster, such as the Herreshoff or the Wedge type, for roasting the crude or the rich ore.

B. Adoption of a special type of reverberatory furnace for the liquation process.

C. Adoption of gas-firing for the reduction furnace.

D. Adoption of the Cottrell electrical precipitation process.

E. Investigation of the possibilities of electric smelting of the oxide or ore.

F. Investigation of the possibilities of the recovery of the sulfur from the fume by means of the thiogen process, as suggested by Prof. Young.

## First Year of Leaching by the New Cornelia Copper Co.

BY HENRY A. TOBELMANN,\* B. S., AND JAMES A. POTTER,† AJO, ARIZ.

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### INTRODUCTION

THE location, mode of occurrence of ore, and preliminary tests leading to the development of the present leaching process and the building of the present plant on the property of the New Cornelia Copper Co. at Ajo, have been described in previous papers presented to the Institute and in the discussions thereof.<sup>1</sup> It is sufficient to say that these tests began early in July, 1912, and were continued to the close of January, 1916; that small tests at Douglas were followed by the construction of a 1-ton plant at the mine; and this was followed by the operation of a 40-ton plant for 11 mo. In all about 15,000 T. of ore were treated experiment-

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<sup>1</sup> Stuart Croasdale: Leaching Experiments on the Ajo ores. *Trans.* (1914) **49**, 610.

Ira B. Joraleman: The Ajo Copper Mining District. *Trans.* (1914) **49**, 593.

L. D. Ricketts: Some Problems in Copper Leaching. *Trans.* (1915) **52**, 737.

H. W. Morse and H. A. Tobelmann: Leaching Tests at New Cornelia. *Trans.* (1916) **55**, 830.

ally during the period mentioned, and those intimately connected with the work came to the conclusion that the process finally developed was right in principle. Accordingly a 5000-ton plant has been constructed and in operation 1 year at the writing of this article. It is proposed here to describe the result of this year's work, the difficulties that have been encountered, and the steps taken to overcome them, and to give an opportunity for those interested to compare the results of the experiments that have been described with the actual operation of a large plant.

The plant was completed on May 1, 1917. The first charge of ore was finished on May 17, and by June 1 the great bulk of solutions in circulation had become sufficiently saturated with copper to permit the operation of the electrolytic plant. On June 18, the first car of cathodes was shipped east to be melted and cast into the finished shapes.

The process adopted was as follows:

1. Mining by steam shovels; the maximum size to be controlled by the size of a fragment that will pass the shovel dipper.
2. Transportation of the ores in cars that will stand up to the rough service and discharge freely any fragments that passed through the dipper of the steam shovel.
3. The delivery of the ore, without any storage other than cars, directly into a crusher that will receive any fragment discharged by the car.
4. Crushing of steam-shovel size to as near  $\frac{1}{4}$  in. as practicable.
5. Leaching the crushed ore 8 days by a counter-current system and upward circulation, using sulfuric acid and such ferric sulfate as is inherent in the process.
6. Reduction by sulfur-dioxide gas of the ferric iron remaining in the neutral solutions from the leaching tanks.
7. The electrolytic deposition of part of the copper from this reduced solution, which is then returned to the leaching solution.
8. The continuous discharge of such portion of the neutral solution as is necessary to prevent accumulation of sulfates, other than copper, to the saturation point.
9. The recovery of the copper content of such discarded solution as cement copper precipitated on iron.
10. The treatment of a part of this cement copper with solution from the electrolytic tank house to the end that the copper be returned to the circulation and a part of ferric sulfate be reduced.

### CRUSHING

The ore is mined by steam shovels and is loaded and delivered to the crushing plant in side dump cars. The crushing plant is divided into two departments, coarse and fine, which are separated by a 10,000-ton storage bin. The coarse-crushing department consists of a No. 24



TABLE 1.—*Summary of Results. May 1, 1917 to May 1, 1918*

Total tons of dry ore charged to leaching plant.....	1,345,000
Total number of tanks charged.....	269
Total copper contents, per cent.....	1.631
Soluble copper contents, per cent.....	1.577
Insoluble copper, probably present as sulfide, per cent.....	0.054
Average proportion of ore on 4-mesh screen, per cent.....	41.9
Average proportion of ore through 20-mesh screen, per cent....	19.1
<hr/>	
Total number of tanks excavated.....	259
Average moisture in tailings, per cent.....	11.1
Total copper in tailings, per cent.....	0.338
Copper in laboratory washed tailings, per cent.....	0.254
Water-soluble copper in tailings, per cent.....	0.084
Average pounds of water-soluble copper per ton of tailings.....	1.6
<hr/>	
Average number of days leached.....	9.7
Average gallons per minute advance through ore.....	1069
Circulation in tank, gallons per minute.....	4500
Average specific gravity of neutral advance.....	1.344
Average free sulfuric acid going on oldest ore, per cent.....	2.85
Average sulfuric acid in solution coming off newest ore, per cent..	0.5
<hr/>	
Average gallons advance per minute through towers.....	1005
Total iron in neutral advance to towers, per cent.....	2.36
Ferric iron in neutral advance to towers, per cent.....	1.06
Ferric iron in neutral advance from towers, per cent.....	0.46
Number of roasters in service.....	3
Average tons of ore roasted per day.....	68.5
Average sulfur contents of ore, per cent.....	42.7
Average sulfur contents of calcines, per cent.....	7.1
Average sulfur dioxide in gas towers, per cent.....	8.1
Average sulfur dioxide in gas from towers, per cent.....	1.9
Average pounds of sulfur consumed per pound of ferric iron reduced.....	0.57
<hr/>	
Average gallons per minute through tank house.....	1005
Average copper content of solution entering tank house, per cent.	3.01
Average copper content of solution leaving tank house, per cent..	2.53
Average copper content removed from solution through tank house, per cent.....	0.48
Average ferric iron contents of solution entering tank house, per cent.....	0.45
Average ferric iron contents of solution leaving tank house, per cent.....	0.99
Per cent. of theoretical oxidation.....	65.6
Average current density, amperes per square foot.....	6.6

Average voltage between anode and cathode.....	2.00
Average weight of cathode shipped, pounds.....	127
Number of tanks on cathodes.....	120
Number of tanks on starting sheets.....	23
Number of starting sheets made.....	266,453
Per cent. of starting sheets scrapped.....	11.4
Pounds of electrolytic copper produced.....	24,400,532
Total kw. hr., A. C. charged to electrolysis .....	34,865,096
Pounds of copper per kw. hr., A. C.....	0.70
Pounds of copper per kw. hr., D. C.....	0.82

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Total tons of acid (60° B. sulfuric acid) charged to plant.....	59,809
Pounds of 60° B. acid per ton of ore leached .....	90.3
Pounds of 60° B. acid per pound of copper dissolved .....	3.56
Pounds of 100 per cent. acid per pound of copper dissolved ....	2.76

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Average pounds of copper dissolved per ton of ore leached .....	26.06
Average per cent. of total copper dissolved.. ..	79.89
Per cent. of total copper into process shipped as electrolytic copper.....	53.70
Per cent. of total copper into process shipped as cement copper..	15.13
Per cent. of total copper tied up in process.....	10.00
Per cent. of total copper produced as electrolytic.....	75.33
Per cent. of total copper produced as cement.....	24.67

---

		PER CENT.
Total pounds of copper to process .....	43,847,000	100.00
Total pounds of copper produced.....	32,392,565	73.88
As electrolytic.....	24,400,532 lb.	
As cement.....	7,992,033 lb.	
Total pounds of copper in solution May 1, 1918.. ..	1,902,768	4.34
Total copper unrecovered,		
In tailings as insoluble copper .....	6,381,242	14.55
In tailings as water-soluble copper .....	2,110,332	4.81
Unaccounted for.....	1,060,907	2.42

gyratory crusher set on a high concrete pedestal. Four No. 8 crushers of the same type, two on each side, receive the product from the large crusher and reduce it to about 4-in. (101 mm.) cubes. This product is taken by two 36-in. (91 cm.) conveyors to the storage bins. The conveyors have magnetic head pulleys; there are also powerful 53-in. (134 cm.) magnets hung over each belt.

The superstructure provides a runway and a 40-ton crane is installed over a No. 24 gyratory crusher for handling parts in repair and for breaking jams in the bowl of the crusher. The 10,000-ton storage bin between the coarse and fine crushers is of steel built on an elevated reinforced-concrete platform. It is flat-bottomed and the ore is drawn

from it automatically onto a set of four 20-in. belt conveyors, equipped with magnetic head pulleys, which deliver the ore to four units of Symons 48-in. vertical-shaft disk crushers. Each unit consists of three crushers, which are interchangeable in every respect. The first is set to crush to inch cubes. The material so crushed is elevated and screened; the undersize by-passes the remaining two crushers, which are set in parallel. The oversize passes to these two crushers which are set to crush to the desired size. The entire product is fed to a system of belt conveyors, which lead through a sampling plant to the leaching vats, which furnish the only storage for the crushed ore. The disk crushers are run by direct-connected 75-hp. alternating-current motors at 400 r.p.m. The pinion shaft is long, permitting the motors to be set in a separate room, thus being protected from dust.

In determining the proper size to which ore should be crushed for leaching, two main ideas are kept in view, crushing for extraction and crushing for percolation. The coarser the product, and the smaller the amount of fines, the freer the circulation will be; but the extraction will tend to vary with the size of the particles. The finer the ore is crushed the higher the extraction will be until such fine crushing materially interferes with the circulation of the solutions. The more uniform the product the better the circulation will be, and, other things being equal, the higher the extraction. The ore used in early leaching tests was crushed by rolls that produced a large amount of fine material. Later a Symons vertical 48-in. disk crusher was used; Symons disk crushers were installed in the 5000-ton plant.

Table 2 gives screen analyses of the ore crushed at Ajo during the first year and represents the analyses on about 1,345,000 T. of ore. The fracture planes of the porphyry are such that the ore has a tendency to break into thin flat pieces, so that the leaching product is better than the screen tests indicate. All fracture planes in the Ajo ore contain more or less malachite and, as a result of crushing, the fine material contains more copper than the coarse. The screen analysis in Table 3, representing a sample from 150,000 T. of ore treated during the month of March, 1918, shows the distribution of the copper values.

The mine is operated in two 8-hr. shifts and the coarse-crushing plant is necessarily operated for the same length of time, there being no storage between the plant and the mine. The fine-crushing plant is run from 3 p. m. to 7 a. m., during which time the charge for a 5000-ton tank is crushed; but as there is a storage bin between the two crushing plants the fine plant can be operated for the full 24 hr. if necessary. Serious trouble has been experienced from the fine dust in the crushing plant. This nuisance has been somewhat abated by wetting down the ore on the cars as it comes from the mine, and a dust-collecting system is now being installed in the fine-crushing plant.

TABLE 2.—*Summary of Screen Sizing Tests for First Year of Operation*

Month	3 Mesh, Per Cent.	4 Mesh, Per Cent.	6 Mesh, Per Cent.	8 Mesh, Per Cent.	10 Mesh, Per Cent.	14 Mesh, Per Cent.	+20 Mesh, Per Cent.	−20 Mesh, Per Cent.
May.....	25.8	18.8	14.2	9.7	7.2	4.1	3.6	16.6
June.....	15.5	18.6	15.7	11.7	8.1	5.5	4.2	20.7
July.....	20.6	18.8	14.9	9.7	7.5	5.2	4.1	19.2
August.....	24.7	16.0	12.2	8.6	7.0	5.3	4.4	21.8
September.....	26.9	17.5	13.2	9.1	6.9	4.9	3.8	17.7
October.....	23.3	17.8	13.9	9.5	7.2	4.5	4.2	19.6
November.....	28.9	16.9	12.4	8.5	7.1	4.9	3.8	17.5
December.....	41.4	13.2	9.7	7.2	5.2	4.1	3.3	15.9
January.....	37.6	12.6	10.8	7.3	6.1	4.5	3.8	17.2
February.....	30.0	14.7	11.9	8.5	6.8	4.8	3.7	19.4
March.....	24.3	15.4	13.9	9.0	7.6	5.6	4.9	19.3
April.....	19.8	17.1	13.5	9.9	7.6	5.6	4.8	21.7
Average.....	26.3	16.6	12.8	9.1	7.0	5.0	4.2	19.0
<i>Extremes—</i>								
Charge No. 144 (1917).....	56.6	9.6	6.6	5.0	4.0	3.1	2.3	12.8
Charge No. 15 (1917).....	7.6	15.2	19.5	14.3	10.1	5.2	5.2	22.9
Charge No. 85 (1917).....	16.1	11.3	12.2	9.9	9.0	7.1	6.0	28.4
40-ton Test Plant:								
Average 290 charges.....	9.7	21.0	20.0	14.1	8.0	6.0	4.2	17.0

TABLE 3.—*Screen Analysis of Ore Treated During March, 1918*

Mesh	Per Cent.	Copper, Per Cent.	Mesh	Per Cent.	Copper, Per Cent.
On 3.....	24.3	1.41	On 10.....	7.6	1.50
On 4.....	15.4	1.35	On 14.....	5.6	1.61
On 6.....	13.9	1.38	On 20.....	4.9	1.71
On 8.....	9.0	1.46	Through 20.....	19.3	2.03

Calculated—1.548 per cent. copper

Actual analyses—1.530 per cent. copper

The ore, as finally crushed, is conveyed by a system of 28-in. (71 cm.) belt conveyors through an automatic sampling plant and thence continuously to the leaching plant. There are twelve leaching tanks arranged in two parallel rows of six each. The aisle between the two rows of tanks is 108 ft. (32.9 m.) wide and contains what is known as the central structure. This so-called central structure consists of six heavy reinforced-concrete piers supporting steel trusses that span from pier to pier. This central structure has two decks, the upper carrying the belt conveyor, the lower, the solution launders and pipe lines. Each unit pier consists of four smaller piers, each supporting a pump and its pipe

connections. Underneath this central structure and parallel to it are two drainage launders used to carry the solutions from the leaching tanks to the solution storage.

### LEACHING

The leaching tanks, 88 ft. (26.8 m.) square and 17 ft. 4 in. (5.28 m.) deep inside, are built of reinforced concrete with wooden bottoms. These have a capacity of 5000 T. each of crushed ore. The walls are 9½ in. (24.1 cm.) thick and are strengthened outside by concrete buttresses. The bottom is of 3 by 8-in. (7.6 by 20.3 cm.) Oregon pine laid edgewise, and is supported by concrete foundations. The sides and launders of the tanks are lined with 8-lb. (3.6 kg.) lead and the bottom with 6-lb. 4-per cent. antimonial lead. The filter bottom is laid over the lead bottom and consists of 5 by 12-in. (12.7 by 30.4 cm.) joists on edge laid on 16-in.

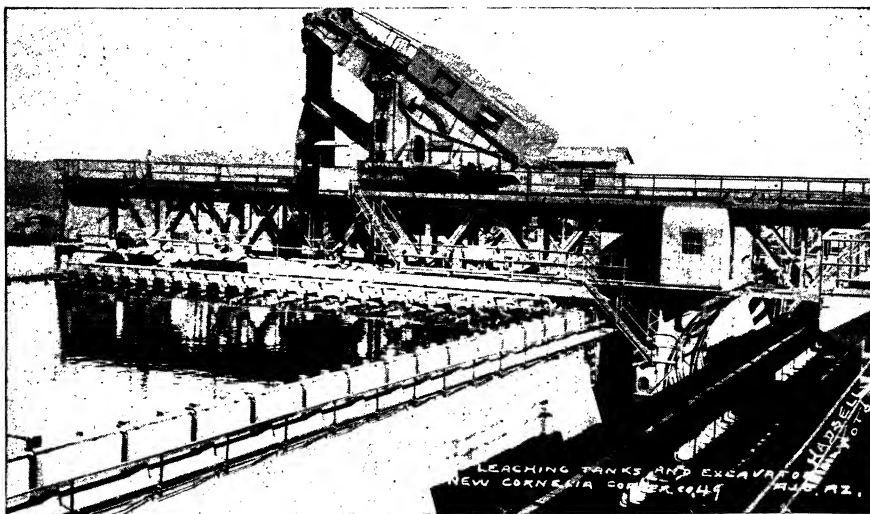


FIG. 1.—LEACHING TANKS AND EXCAVATOR, NEW CORNELIA COPPER CO.

(40.6 cm.) centers, covered with 2-in. ship-lap planks that are bored with ⅜-in. (9.5 mm.) holes on 2-in. (50.8 mm.) centers countersunk from below. Under the center of the filter bottom, and at right angles to the wooden floor joists, there is a distributing launder 5 ft. (1.5 m.) wide by 2 ft. 9 in. (0.8 m.) deep, set in the floor through which the solution enters and from which it is distributed under the filter bottom. The lead lining on the sides of the tanks is protected from abrasion by a covering of 2-in. planks held in place by 6 by 8-in. (15.2 by 20.3 cm.) vertical posts clamped at the bottom and top. At the top and sides of each leaching tank are two overflow launders extending the length of the

tank, one end being connected with the suction of a circulating pump. The charging is done by means of a machine especially designed for this plant and known as the spreader bridge; it consists of a traveling bridge of structural steel which spans the tanks and travels, as desired, lengthwise with the row. The bridge supports a 28-in. (71.1 cm.) belt conveyor, which receives the crushed ore from the belt on the central structure. A tripper on this belt spreads the ore in the leaching tanks.

At the present time the tanks are charged by filling to the top of the tank at one side, allowing the ore to assume its natural slope, or about  $45^\circ$ , and then continuing at one side and discharging the ore at the top of the slope, allowing the coarser material to run to the bottom and the finer to remain somewhat higher up, thus giving a rough classification.

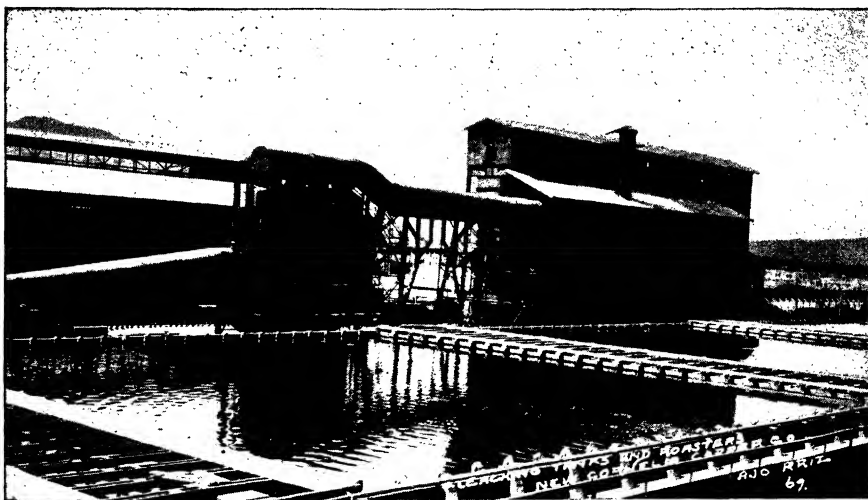


FIG. 2.—LEACHING TANKS AND ROASTERS, NEW CORNELIA COPPER CO.

The bridge is moved slowly forward as the filling of the tank progresses. This plan was suggested by the engineers of the Chile Copper Co., who have obtained the best results through this method of filling.

The crushed ore is leached from six to eight days by a counter-current system and upward percolation, using dilute sulfuric acid as the principal solvent of the oxidized copper minerals.

The solution in each tank is circulated by two 15-in. (38.1 cm.) vertical-centrifugal pumps of 3500 gal. (13,248 l.) per min. capacity each. These are driven by direct-connected 40-hp. vertical motors. The head against which the pumps work is 28 ft. (8.5 m.), which is virtually equivalent to the friction head of the solution passing through the ore. The discharge from one of these pumps is provided with a by-pass which permits a portion of the solution to be advanced to the next tank. Both pumps are

throttled to give a circulation of about 4000 to 5000 gal. per min. through the ore. Of this amount about 1000 gal. (3785 l.), called the solution advance, is continuously passing through from tank to tank. The high-acid solution (3 per cent. free sulfuric acid) coming from either or both the tank house and the solution storage and going on the oldest ore, is called the "acid advance." The nearly neutral solution coming off the newest charge and going to the reduction towers is known as the "neutral advance."

### *Cycle of Leaching Operation*

To understand more clearly the various operations taking place during a leach cycle, the accompanying flow sheet for a day's operation should be followed. The arrangement of the piping and launders permits the advance of solution in but one direction, clockwise. The tanks are also charged in this order. Of the twelve tanks, eleven are used as leaching tanks and one as a solution settler. Of the eleven tanks, seven always contain ore in the process of leaching. Referring to flow sheet, Fig. 4, if we assume the ore in tank 10 to be the oldest and that in tank 5 to be the newest in circuit, then tank 6 is being charged with ore, tank 7 is empty, tank 8 is being excavated, and tank 9 is in the various stages of washing and draining.

When the charging of tank 6 has been completed and the tank is ready for the leaching cycle, the acid advance is increased to the maximum (2000 gal. per min. for 4 hr.) by the addition of solution from the storage tanks A or E. Meanwhile the usual advance of about 1000 gal. per min. continues to go from tank 5 to the reduction towers and the excess solution is advanced to tank 6 until the ore is covered; this is to prevent any interruption in the solution flow to the towers. When the ore is covered this additional advance is cut off and the normal advance resumed. The solution on the new charge is now circulated on itself for about 4 hr., or until clarified. Tank 6 is now put into circuit and the neutral advance to the towers comes off tank 6 in place of tank 5.

The leaching of the ore in tank 6, now begun, continues for 7 days, during which period the free acid in the solution has varied from 0.5 per cent. on the first day to 3.0 per cent. on the seventh. To show the acid concentrations during a leach cycle, the daily analyses of the solution going on each day were averaged for some 18 consecutive charges. The results were as follows:

	PER CENT.
Free $\text{H}_2\text{SO}_4$ going on the ore the seventh day.....	3.0
Free $\text{H}_2\text{SO}_4$ going on the ore the sixth day.....	2.6
Free $\text{H}_2\text{SO}_4$ going on the ore the fifth day.....	2.2
Free $\text{H}_2\text{SO}_4$ going on the ore the fourth day.....	1.9
Free $\text{H}_2\text{SO}_4$ going on the ore the third day.....	1.6
Free $\text{H}_2\text{SO}_4$ going on the ore the second day.....	1.2
Free $\text{H}_2\text{SO}_4$ going on the ore the first day.....	0.9

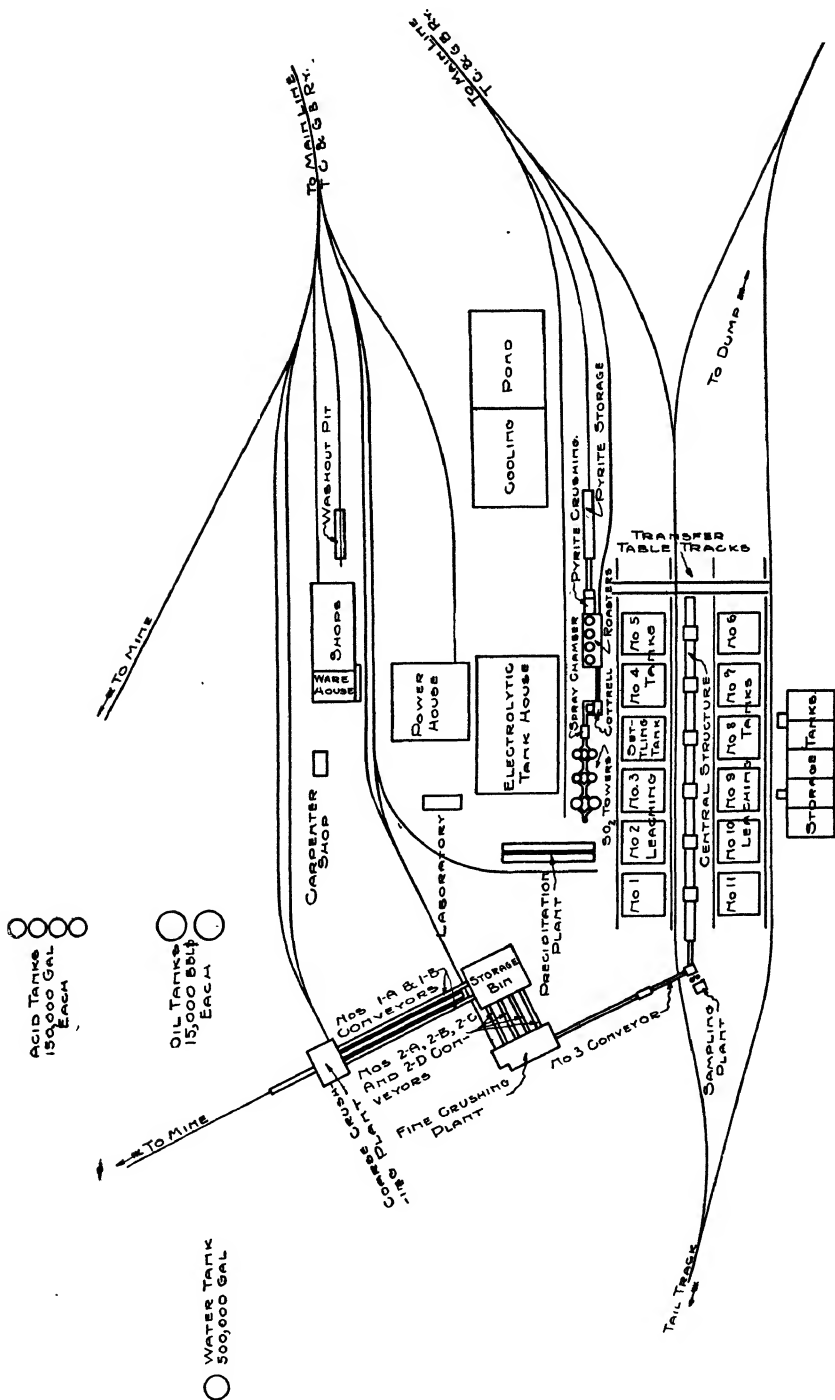


FIG. 3.—GENERAL PLAN OF PLANT.



At the end of the seventh day, the acid advance from the tank house is transferred from tank 10 to tank 11. Upon the entrance of a new charge into the circuit the solution remaining in the oldest tank is drained to the solution storage, where it is standardized with acid and is later used as acid advance. After thorough draining, the tank is ready for the wash water.

As the copper entrained in a charge after leaching is about one-third of the total copper dissolved, the question of thorough washing is very important. Four successive wash waters with drainings are used. During the 3 hr. circulation that each wash is given, an equilibrium between the dissolved copper in the tailings and that of the wash water being applied is expected to be reached. To follow more readily the method of washing a charge, the flow sheet is referred to. When tank 9 has been thoroughly drained, the charge is covered with wash water from wash-water tank 1, circulated, and then drained to solution storage tank A or E; this constitutes the first wash. The charge is now covered with wash water from wash-water tank 2 and similarly circulated, and then drained into wash-water tank 1. In the same manner, the wash water from wash-water tank 3 is put on, circulated, and drained. The fourth or last wash, consisting entirely of fresh water, is pumped, circulated, and drained into wash-water tank 3.

In this manner the fourth wash of any one charge is used as the third wash of the succeeding charge, the third used as the second, and the second as the first. In other words, each wash water is used four times, the copper contents increasing each time, when it is incorporated into the system to make up the continuous losses of solution. These losses are due to evaporation, discard, and solution entrained in tailings.

The average analyses of the wash waters for March, 1918, were as shown in Table 4.

TABLE 4.—*Analyses of Wash Waters for March, 1918*

	Solution Off	First Wash	Second Wash	Third Wash	Fourth Wash
Free H <sub>2</sub> SO <sub>4</sub> , per cent.....	2.56	1.02	0.68	0.48	0.10
Copper, per cent.....	2.40	1.61	1.15	0.74	0.38
Ferrous iron, per cent.....	1.58	1.06	0.70	0.47	0.04
Ferric iron, per cent.....	0.72	0.48	0.31	0.17	0.26
Specific gravity.....	1.30	1.20	1.13	1.08	1.05

To obtain the best results in washing a charge, the tanks should be thoroughly drained after each wash and the circulation should be limited by the time required to reach an equilibrium. As each 0.1 per cent. of copper in the last wash water means a loss of over 1000 lb. of dissolved copper

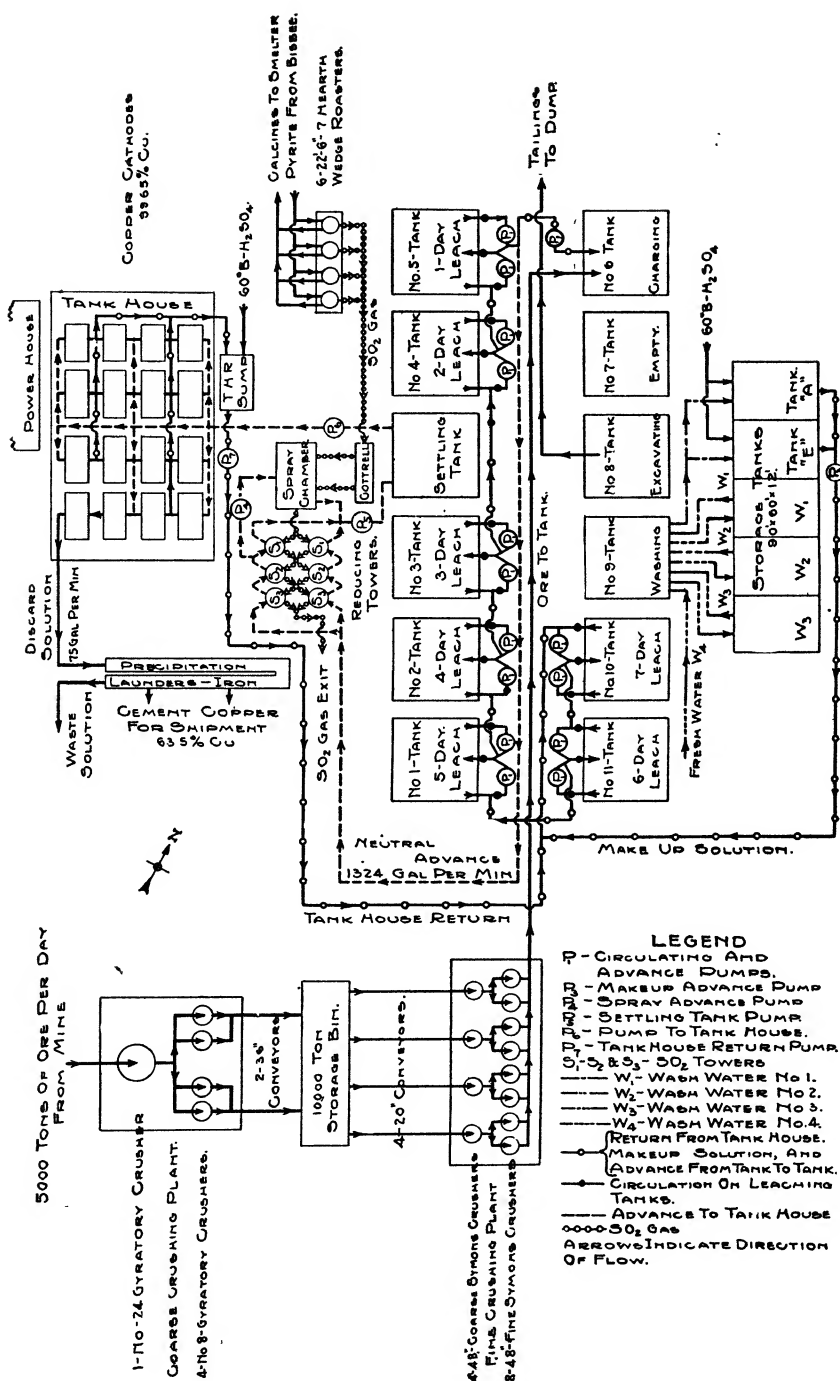


FIG. 4.—GENERAL FLOW SHEET OF ORE AND SOLUTION.

per day, it is evident that the washing has not yet been perfected. Limited solution storage and launder installation during the first year has made the use of a fifth wash water impracticable. Probably the best recommendation made in connection with the use of a fifth wash water was the precipitation of its copper contents on scrap iron simultaneously with its use, using the same water repeatedly until it reaches a point where it is no longer effective. Plans are now being considered for this installation. The losses that the wash waters replace are shown in Table 5. The total is about the volume of one wash water. The average moisture in the tailings for the first year of operation was 11.12 per cent.

TABLE 5.—*Losses of Solution*

	Gallons per Ton of Ore	Gallons per Day
Evaporation.	5 to 10	25,000– 50,000
Discard.....	28 to 30	140,000–150,000
Entrained in tailings.	24 to 26	120,000–130,000
Total.....	57 to 66	285,000–330,000

#### *Removal of Tailings*

After a charge has been washed and drained, the tailings are removed from the tank by means of a Hulett unloader. The machine was furnished by the Wellman-Seaver-Morgan Co., and is similar to unloaders used on the Great Lakes for unloading iron ore from boats. A heavy steel bridge on trucks spans the leaching tanks and travels their entire length. On this bridge travels a trolley carrying a walking beam, bucket leg, and bucket of 12 T. capacity. The unloader has a rated capacity of 500 T, per hr. and will generally excavate a tank of tailings in 10 to 11 hr. An engineer and an oiler are required to operate it. A description of this machine has been written by Franklin Moeller, Engineer for the Wellman-Seaver-Morgan Co.<sup>2</sup> Two eight-car trains are released from the mine service at 4.30 p. m. for the transportation of tailings. Twenty-one to twenty-three train loads of eight cars each are required to excavate a tank. The tailings are taken to a dumping ground located about a mile from the plant. The dump is on gently sloping ground with a uniform grade of 1 per cent., the end of the present dump being about 80 ft. (24.3 m.) above the original ground.

#### *Solution*

The acid advance during the first year has varied from 869 to 1324 gal. (3289 to 5011 l.) per min. While we found that the more rapid

<sup>2</sup> *Trans.* (1919) 61.

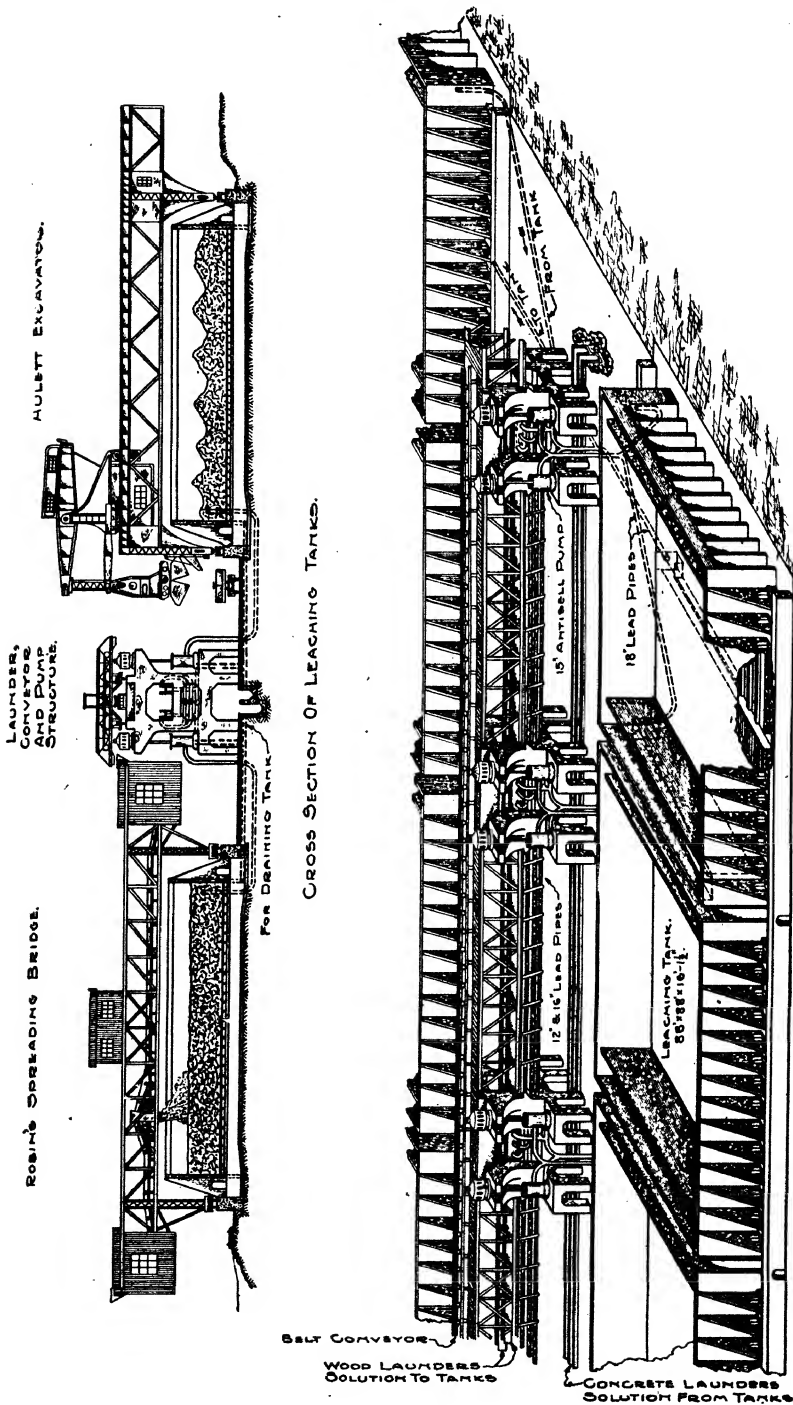


FIG. 5.—CROSS-SECTION AND CAVALIER PROJECTION OF LEACHING TANK.

advance appeared to give better extractions, we also found that the acid consumption was greatly increased. It is of interest to note that the per cent. of free acid coming off the newest charge averaged practically the same for an advance of 869 gal. per min. as for an advance of 1324 gal. per min. To show the normal decrease in acid and increase in copper and other constituents in the solution advance in one passage through the leaching tanks, the average analyses for the month of March, 1918, are given in Table 6.

TABLE 6.—*Analyses of Solution for March, 1918*

	Acid Advance	Neutral Advance
Free sulfuric acid, per cent.....	3.00	0.48
Copper, per cent.....	2.38	3.04
Iron as ferrous iron, per cent.....	1.57	1.61
Iron as ferric iron, per cent.....	0.69	0.72
Alumina, per cent.....	2.53	2.60
Specific gravity.....	1.290	1.305

There were originally some 13,000,000 T. of thoroughly oxidized ore, consisting principally of malachite together with small amounts of cuprite and chrysocolla distributed through a monzonite-porphyry gangue. Of this tonnage over 10 per cent has already been treated. It came in nearly equal quantities from three widely separated parts of the orebody. From this it is reasonable to believe that equally good or better results may be expected from the remainder. The ore so far mined has been better than the sampling of the property indicated, averaging 1.631 per cent. total copper as against 1.54 per cent. reported. Occasionally small quantities of sulfides are encountered, but not enough to make it worth while to treat this ore separately. Both the total and the soluble copper content of each charge of ore bedded is determined. The difference between the total copper and the soluble copper is an indication of the quantity of copper present as sulfides; for the first year of operation it was 0.054 per cent.

Comparing the analyses and assays on corresponding composite samples of the ore before and after leaching, it is found that a measurable quantity of iron, alumina, and magnesia has been dissolved. Table 7 gives the analyses of the ore before and after treatment during February, 1918. A study of the table will show relative solubilities of the various constituents of the ore in our present leaching solution. This was one of the first things to be determined in developing this process. Mr. Croasdale showed that the quantity of soluble material other than copper is comparatively small. Furthermore, comparison of the sizing tests of the ore before and after treatment shows that no appreciable physical

TABLE 7.—*Analyses of Ore Before and After Leaching, February, 1918*

	Heads, Per Cent.	Tails, Per Cent.		Heads, Per Cent.	Tails, Per Cent.
SiO <sub>2</sub> .....	67.04	69.28	P <sub>2</sub> O <sub>5</sub> .....	0 13	0.110
Fe, total.....	5.05	4.33	Na <sub>2</sub> O.....	1.73	1.60
Al <sub>2</sub> O <sub>3</sub> .....	12.30	11.50	K <sub>2</sub> O.....	3.34	3.31
CaO.....	0.63	0.60	TiO <sub>2</sub> .....	0 44	0.48
MgO.....	1.52	1.24	CaO as CaSO <sub>4</sub> .....		0.27
Mn.....	0.025	0.02	Fe as ferrous iron.....	3 57	2.05
S.....	0.05	0.26	Fe as ferric iron.....	1.48	2.28
S (sol. in H <sub>2</sub> O).....		0.16	H <sub>2</sub> O.....	0 95	0 93
Cu, total.....	1.57	0.28	Au, ounces per ton.	0 014	0 014
Cu, (sol. in H <sub>2</sub> SO <sub>4</sub> )....	1.51		Ag, ounces per ton	0 161	0.157
Cu, in laboratory washed tailings.....		0.22			

change has taken place as a result of the solution of these constituents.

A part of the regular daily laboratory work consists of making analyses of a composite of hourly samples of the solutions on and off all leaching tanks. An average of the analyses of 18 consecutive charges is shown in Table 8. The results proved so interesting that 94 charges were similarly

TABLE 8.—*Composite Analyses of Solutions on and off Tanks*

	Gain in Copper Concentration, Per Cent.	Drop in Acid Concentration, Per Cent.	Converted Pounds per Ton of Ore Treated	
			Pounds of Copper Dissolved	Pounds of 60° B H <sub>2</sub> SO <sub>4</sub> Consumed
1st day.....	0 094	0 194	3 66	9.74
2d day.....	0.138	0 288	5.37	14.45
3d day.....	0.111	0.318	4.32	15.90
4th day.....	0.108	0 388	4.58	19.40
5th day.....	0.098	0.278	3.81	13.85
6th day.....	0 063	0.350	2.45	17.55
7th day.....	0 015	0.394	0.58	19.60
8th day*.....	0 032	0.394	1.24	19.60
Total.....	0.659	2.604	26.01	130.09

\* The eighth day represents the maximum acid solution.

averaged, including some charges that for various reasons were permitted to remain in the leaching tanks for 10 days. The results for the first eight days on these 94 charges checked very closely those of the 18 eight-day charges; therefore, a comparison will be made only

of the last four days of the leaching period. From this, it is obvious that the 0.7 lb. of copper gained per ton of ore leached on the last two days was obtained at a cost of nearly 43 lb. (19.5 kg.) of 60° B. sulfuric acid,

TABLE 9.—*Copper Dissolved and Acid Consumed*

Number of Charges Averaged	Pounds of Copper Dissolved per Ton of Ore Treated		Pounds of 60° B. Acid Consumed per Ton of Ore Treated	
	94	18	94	18
7-day leach .....	25.7	24.8	109.2	112.7
8-day leach .....	26.9	26.0	129.6	130.0
9-day leach .....	27.3		150.9	
10-day leach .....	27.6		172.2	

and that there is a well-defined point where a high extraction is not profitable, if it is obtained by prolonging the time of contact between ore and acid. The pounds of material dissolved per ton of ore leached and the pounds of acid consumed appeared to be as shown in Table 10.

TABLE 10.—*Material Dissolved and Acid Consumed*

	Number of Days Leached									
	1	2	3	4	5	6	7	8	9	10
Cu, lb. per ton ore leached.....	3.60	8.75	14.50	19.11	22.47	25.00	26.72	27.93	28.26	28.65
Fe, lb. per ton ore leached.....	0.00	0.00	0.00	0.98	2.28	4.62	5.70	6.80	8.42	9.22
Al <sub>2</sub> O <sub>3</sub> , lb. per ton ore leached.....	0.00	0.17	1.69	1.40	2.29	2.76	4.61	7.33	10.22	13.65
MgO, lb. per ton ore leached.....	0.00	0.08	0.58	0.63	1.03	1.24	2.08	3.31	4.60	6.15
Pounds of 100 per cent. acid used per ton of ore.....	8.1	19.1	32.4	47.2	59.4	71.7	84.8	100.6	117.2	133.8

Leaching tests made on individual screen sizes in a small lead Pachuca tank holding approximately 200 lb. (90 kg.) of ore showed the rates at which the various impurities dissolve compared to that of copper. The ore was carefully screened, dried and weighed. The acid was weighed and the solution measured. The acid concentration was kept as near as was possible to that of the 5000-ton plant for the corresponding day. So far, tests have been completed on three screen sizes: Minus 0.525 in. plus 0.371 in., minus 3 mesh plus 4 mesh, and minus 6 mesh plus 8 mesh. The cumulative pounds of material dissolved per ton of ore leached are as follows:

TEST 1.—*Minus 0.525 Plus 0.371-mesh Screen*

	Hours Leached					
	24	48	72	96	120	144
Cu.....	10 34	14.36	16.97	18.82	21.22	22.14
Fe.....	0.43	0.79	1.21	1.64	2.03	2.57
Al <sub>2</sub> O <sub>3</sub> ...	1 04	1.71	2.19	2.64	3.26	3.36
MgO....	0.23	0 31	0.35	0 40	0 70	0.80
CaO....	1 19	1.36	1.18	1 22	1.19	0.98
P <sub>2</sub> O <sub>5</sub> ....	0 25	0 35	0 44	0 47	0.63	0.64

TEST 2.—*Minus 3-mesh Plus 4-mesh Screen*

	Hours Leached						
	13	30	48	72	97	125	144
Cu... . . . .	14 80	18 30	21.10	22.30	24.10	25 00	26 60
Fe ..	0 65	0 89	1 29	1.74	2.16	2 96	3.96
Al <sub>2</sub> O <sub>3</sub> ...	1 18	1 99	2.47	2.46	2 79	3 71	4.08
MgO..	0 25	0.32	0 34	0 39	0.49	0 73	0 90
CaO... .	1 52	1.15	1.08	1 01	0 92	1.05	1 08
P <sub>2</sub> O <sub>5</sub> .	0 34	0 47	0 58	0 66	0 85	1 05	1.03

TEST 3.—*Minus 6-mesh Plus 8-mesh Screen*

	Hours Leached				
	12	24	36	60	72
Cu.....	18 20	21 10	22 50	22 60	23.40
Fe .....	0 70	1 00	1.15	1.55	1.85
Al <sub>2</sub> O <sub>3</sub> .....	1 69	2 28	2 59	2.88	3 01
MgO.....	0.26	0 34	0.37	0 31	0 42
CaO.....	1 27	1 18	1 08	1.08	1 08
P <sub>2</sub> O <sub>5</sub> .....	0.25	0 37	0 43	0 76	0 83

Calculating these results so as to show the solubility of impurities, in pounds per pound of copper dissolved, we have Table 11. These results indicate that fine crushing gives the highest extraction in the least time and with the minimum acid consumption.



TABLE 11.—*Impurities Dissolved per Pound of Copper*

	24-hr. Leach			72-hr. Leach			144-hr. Leach	
	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3	Test 1	Test 2
Fe.....	0.042	0.043	0.044	0.071	0.078	0.079	0.116	0.162
Al <sub>2</sub> C <sub>3</sub> .....	0.109	0.080	0.108	0.129	0.110	0.129	0.152	0.166
MgO.....	0.021	0.016	0.016	0.021	0.017	0.018	0.036	0.037
CaO.....	0.115	0.102	0.060	0.069	0.045	0.046	0.044	0.044
P <sub>2</sub> O <sub>5</sub> .....	0.024	0.023	0.017	0.026	0.029	0.035	0.029	0.042
Total copper dissolved, per cent..	31.6	55.6	80.0	51.9	74.9	91.5	67.7	89.2
Pounds of acid consumed per lb. of copper dissolved.....	2.0	2.0	2.4	2.3	2.2	2.7	2.4	2.6

*Acid Consumption*

Upon the amount of acid consumed per pound of copper may depend largely the profit to be derived from the ore. During some 300 charges, the average acid consumption for the 40-ton plant was 2.8 lb. (1.3 kg.) of 100 per cent. acid per pound of copper dissolved. This does not include that part regenerated in the tank house by the electrolytic deposition of copper or that obtained by the oxidation of the SO<sub>2</sub> to H<sub>2</sub>SO<sub>4</sub> by the ferric iron. Mr. Croasdale's experiments showed a probable acid consumption of about 3.15 lb. (1.4 kg.) of 100 per cent. H<sub>2</sub>SO<sub>4</sub> per pound of copper dissolved. This was of course the net consumption, as there was none produced or regenerated.

The amount of new acid introduced into the system during the first year has varied from month to month. By new acid we mean that part of the acid that must be brought in from an outside source, and represents only about 60 per cent. of that actually consumed in dissolving the copper in the ore; of the remaining 40 per cent., 32 per cent. is that generated in the towers by the oxidation of SO<sub>2</sub> and 8 per cent. is that regenerated in the tank house by the deposition of copper. In the final report on the experimental work, we stated that this acid consumption on the 5000-ton plant would not exceed 3 lb. of 100 per cent. H<sub>2</sub>SO<sub>4</sub> per pound of copper dissolved.

For the first 5 mo. of the year, both the development work at the mine and the usual troubles in starting a plant prevented the delivery of 5000 T. of ore per day. This condition resulted in many of the charges being in process from 10 to 16 days, during which time great quantities of acid were consumed and but little copper extracted. For the last 7 mo. these conditions have been continually bettered and the average acid consumption for the first year of operation is 2.76 lb. (1.25 kg.) of 100 per cent. H<sub>2</sub>SO<sub>4</sub> per pound of copper, or 3.36 lb. (1.5 kg.) of 60° B. acid per pound of copper, while the average for the last 4 mo. is 2.75 lb. (1.24 kg.) of 60° B. or 2.14 lb. (0.96 kg.) of 100 per cent. acid.

Approximately 60,000 T. of 60° B. sulfuric acid have been charged out to the leaching process during this first year of operation, or an average of 164.4 T. per day. During this period, some 1,345,000 T. of ore have been put into process, making an average acid consumption of 89.6 lb. (40.5 kg.) of 60° B. acid per ton of ore. The acid used is purchased from the Calumet & Arizona Mining Co. acid plant at Douglas, Ariz., and is delivered to Ajo, some 300 miles distant, in 50-ton iron tank cars at the rate of about three cars a day. Ample storage has been provided by the erection of four 1000-ton steel tanks, which permit of keeping 3500 to 4000 tons of acid in stock. A 6-in. (15 cm.) pipe line delivers the acid by gravity to the electrolytic tank house and to the solution storage.

Since the Ajo ores carry neither arsenic nor antimony, which are very deleterious impurities in cathode copper, the only other source of introduction for these impurities would be sulfuric acid. As the ore used for the production of the acid at Douglas carries but very small quantities of these impurities, the acid is of good quality, and there is no trouble from this source.

TABLE 12.—*Analysis of Sulfuric Acid (Oct. 1, 1917 to Jan. 1, 1918)*

	Per Cent.		Per Cent		Per Cent.
H <sub>2</sub> SO <sub>4</sub> .....	80.86	Cu.....	0 0618	Fe.....	0 0219
	(Equivalent	Pb.....	0 0032	Zn.....	0 0161
	to 61.5° B.)	Sb.....	0 0063	Bi.....	None
N <sub>2</sub> O <sub>3</sub> .....	0 0064	As . . . . .	0 0058	Cl.....	None
HNO <sub>3</sub> .....	None				

An analysis of three months' composite sample of the acid is shown in Table 12. The acid occasionally has a pinkish tint, which upon examination was found to be due to a trace of selenium. After operating several months, a blood-red scum appeared in spots on the oil on the surface of the electrolyte. Anodes withdrawn at about that time showed a similar coating on the porcelain insulators. This scum or deposit was tested and found to be selenium.

To show how the acid is consumed, a typical solution analysis is given in Table 13. This is the analysis of a composite sample representing solution from the whole leaching and electrolytic cycle on Dec. 27, 1917. The specific gravity is 1.319. There are about 6,000,000 gal. (22,710,000 l.) or approximately 30,000 to 35,000 T. of such solution on hand at all times throughout the plant, representing a tie-up on the average of some 900 T. of copper and 500 T. of acid (100 per cent.). Large

as this may appear it is believed that it will not equal the quantity of copper that is tied up at the average smelter with an equal production.

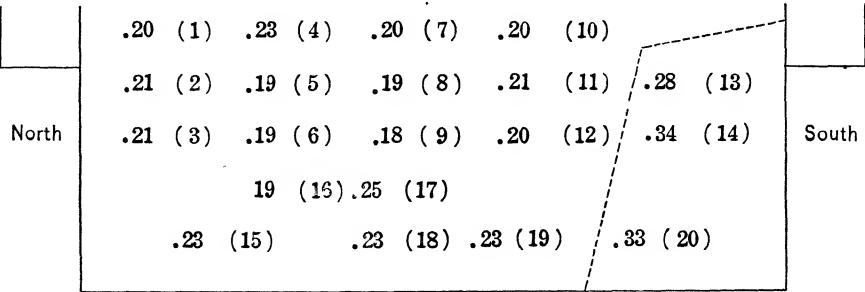
TABLE 13.—A Typical Solution Analysis

	Per Cent.		Per Cent.		Per Cent.
Cu.....	2.736	P <sub>2</sub> O <sub>5</sub> .....	0.192	H <sub>2</sub> SO <sub>4</sub> , free .	1.500
Al <sub>2</sub> O <sub>3</sub> .....	2.758	CaO.....	0.055	As. ....	0.003
Fe, total.....	2.456	MnO... ..	0.038	Sb... ..	0.002
Fe as ferrous....	1.511	Cl... ..	0.012	Zn .. ..	0.033
Fe as ferric.....	0.945	SO <sub>3</sub> .. ..	17.004	Se .. ..	Trace
MgO.....	1.420				

The free acid in the solution going through the ore has been maintained at practically a constant percentage during the first year, but on comparing the per cent. of copper in the tailings of charges of like size and equal time of contact with the solution, wide variations are noted. No reason could be found for many of these variations.

To determine whether the copper contents of the tailings in the different parts of a tank are uniform, several tanks were sampled. The results on two of these tanks are shown. Each train-load was sampled separately. The tailings were sampled by taking eight 8-oz. (178 gm.) scoops full from the top of each car in predetermined places. The location in the cross-section of the tank from which the different samples were drawn was recorded. Since there was a difference in the per cent. of coarse and fine material in the different parts of the tank, the amount of copper was determined only in the tailings between 3 and 4 mesh. Figures in parenthesis are the cut number, while the others give the per cent. copper in the samples from these cuts.

Charge 61, Tank 6.—This tank was charged by filling one side to the top and then advancing from the south to the north side, as shown below.



Tank charged, March 10, 2.10 p. m. Solution on, March 10, 6.30 p. m. Solution off, March 17, 4.00 a. m. Total time of leaching, 6 days, 9 hr., 30 min. Excavating began, 5 p. m. Finished, 4.00 a. m. Time, 11 hr. The last train-load was a general clean-up of the floor and showed 0.20 per cent. copper.

	COPPER, PER CENT.
Average of above 21 train-loads, 4 mesh.....	0.223
Regular tailings sample, 4 mesh.....	0.24
Regular tailings sample, all mesh.....	0.24

Sizing Test, Mesh	3	4	6	8	10	14	+20	-20
Tailings								
On, per cent.....	23.80	21.90	13.30	9.10	7.50	4.50	3.80	16.10
Cu, per cent.....	0.44	0.24	0.15	0.14	0.14	0.15	0.14	0.22

Regular head sample, per cent. copper.....	1.51
Copper dissolved, per cent.....	84.1

*Charge 127, Tank 6.*—The tank was charged by filling both sides to the top and then advancing from the south to the north side, as shown in the following diagram.

North	.27 (1)	.23 (4)	.31 (7)	.24 (10)	.29 (13)	South
	.28 (2)	.22 (5)	.27 (8)	.25 (11)	.37 (14)	
	.27 (3)	.23 (6)	.24 (9)	.23 (12)	.31 (15)	
	.21 (22)	.22 (20)	.23 (18)	.25 (16)		
	.22 (25)	.21 (23)	.20 (21)			
	.20 (24)		.25 (19)	.28 (17)		

Tank charged, May 14, 8.10 a.m. Solution on, May 14, 11 p. m. Solution off, May 21, 3.30 p. m. Total time of leaching, 6 days, 4 hr., 30 min. Excavating began, 5.20 p. m. Finished, 3.30 a. m. Time, 10 hr. 10 min.

The space within the dotted lines represents the zone of highest circulation and hence the most intense leaching action. The close agreement between the average of all the segregation assays and that of the regular tailings sizing test on similar sized material indicates consistent sampling. This is further emphasized by a comparison of the head sizing test (automatic sample) and the tailings sizing test, which is a hand sample.

	COPPER, PER CENT.
Average of 25 train-loads, assay of 4-mesh material.....	0.25
Regular tailings sample.....	0.25
Sizing test on regular sample, 4 mesh.....	0.27
Total copper calculated from sizing test.....	0.25

Sizing Test, Mesh	3	4	6	8	10	14	+20	-20
Heads								
On, per cent.....	19.60	17.30	15.00	10.40	7.60	5.50	4.20	20.40
Cu, per cent.....	1.29	1.30	1.24	1.27	1.34	1.43	1.44	1.86
Tailings								
On, per cent.....	14.70	20.40	14.60	10.00	8.40	5.40	4.40	22.10
Cu, per cent.....	0.38	0.27	0.21	0.18	0.19	0.19	0.18	0.27

Regular head sample, per cent. copper.....	1.36
Calculated copper from sizing test, per cent. copper.....	1.42
Copper dissolved, per cent.....	81.60

*Effect of Variation in Circulation*

Charges were run with various rates of circulation, but no decided effect could be noted. Samples of ore taken from the top of a charge at regular intervals showed that where the solution had free access to the ore only 3 days were required to dissolve 80 per cent. of the copper in the ore, as against 6 days for the whole charge. These results and others lead to the belief that the crushing and the bedding of a charge so as to give the best circulation is of prime importance. In other words, the greatest care should be exercised in bedding and in regulating the circulation so as to prevent channeling of the solution. Upward circulation was selected because it reduces the tendency toward channeling and also effects more rapid solution of the copper.

Results obtained show that small variations in the density have no appreciable effect on the solubility of copper in the solution. In spite of the self-evident fact that a light solution is a more active lixiviant than a heavy one, a certain density must be maintained to keep the remainder of the process in balance.

At the beginning of operations the lead lining of the leaching tanks was exposed to the channeling of the ore and serious abrasion was noticed. The ore buoyed up by the heavy solution circulating at a high rate had completely worn through the lead in places. This condition was remedied by lining the tanks with 2 by 12-in. (5 by 30 cm.) planks and no further trouble has been experienced from this source. The solution was prevented from coming up between the plank and the lead lining by calking with oakum and asphalt. The bottoms of the leaching tanks are lined

with 4-per cent. antimonial lead, while the sides are of chemical lead. Some trouble was at first experienced from splitting at the seams, between the hard and soft lead lining. This was caused by expansion, contraction, and defective burning. These seams were repaired and no further trouble was experienced. There were no important changes made either in the equipment or operation of the leaching division. The only change of interest was the reduction in the time of leaching from the original 8-day period to that of 6 to 7 days.

### *Water Supply*

At the time the property was taken over, the nearest known water supply in sufficient quantity for our purpose was the Gila River, some 45 mi. (72.42 km.) to the north. The development of a closer water supply was one of the important problems to be solved. A large valley about 8 mi. (12.875 km.) northeast of Ajo was selected as a likely place and two wells were drilled. In both water was found at a depth of about 600 ft. (182 m.) having a temperature of 104° F. At one of these places a two-compartment shaft was put down and a modern pumping plant installed. This shaft is now delivering 800,000 to 1,000,000 gal. (3,028,000 to 3,785,000 l.) of water per day, without any sign of decrease in volume. The pumping installation consists of a duplex double-acting high-pressure pump driven by a direct-connected synchronous motor. The water is pumped 6.7 mi. (10.7 km.) through a 10-in. (25 cm.) iron pipe line against a 1375-ft. (419 m.) head, including friction.

The sinking of another well, about a year later, by a rancher, some 6 mi. east of Ajo and about 12 mi. from the present well, proved the extent of the water course. The water found was of practically the same temperature and analysis. This made reasonably certain a continuous and plentiful supply. In this connection a comparison of the analyses of the water at these wells will be of interest. The analyses were made according to a well-known method of boiler-water analysis and are therefore comparable, see Table 14. Samples 1 and 2 were taken during development of the present supply. The analyses were made in the Calumet & Arizona laboratory, Bisbee, Ariz. Sample 3 was taken from plant tap near the end of the construction period, after pumping approximately 100,000,000 gal. from the well; sample 4 was taken after pumping approximately 400,000,000 gal., and sample 5 is water from the ranch well southeast of Ajo. The water from our main well is used for all purposes, including leaching plant, mine, railroad, and townsite. The great depth of the well and its distance from any possible sources of contamination make it an unusually good potable water. Further drifting in the present well would no doubt increase the flow should more water be required.

TABLE 14.—*Analyses of Water in Grains per U. S. Gallon*

	Sample 1 4-15-14	Sample 2 5-18-15	Sample 3 11-16-16	Sample 4 4-15-18	Sample 5 3-20-17
SiO <sub>2</sub> , insoluble.....	3.90	2.71	2.28	2.52	2.22
Fe <sub>2</sub> O <sub>3</sub> + 1Al <sub>2</sub> O <sub>3</sub> .....	0.17	0.11	1.14	0.05	0.37
CaCO <sub>3</sub> .....	3.00	2.20	1.95	1.90	2.10
CaSO <sub>4</sub> .....	none	none	none	none	none
CaCl <sub>2</sub> .....	none	none	none	none	none
MgCO <sub>3</sub> .....	0.73	0.71	0.84	0.80	1.13
MgSO <sub>4</sub> .....	none	none	none	none	none
MgCl <sub>2</sub> .....	none	none	none	none	none
Na <sub>2</sub> CO <sub>3</sub> .....	4.34	4.21	4.95	5.04	4.71
NaSO <sub>4</sub> .....	8.52	7.78	7.70	7.66	8.99
NaCl.....	16.93	16.76	19.30	14.83	19.11

## REDUCTION

In the electro-deposition of copper from a sulfuric-acid solution, iron if present will consume electric energy, by its alternate oxidation and reduction, thereby reducing the quantity of copper deposited per unit of current. During the experimental work the control of the ferric iron proved one of the hardest problems to solve. A patent diaphragm-anode was tried and gave good results, but was cumbersome and difficult to keep in order. Later, tests made on a process in which iron and alumina were precipitated as hydrated oxides by the addition of roasted copper ores gave good results. This method was considered too complicated for an ore of this character. The idea was then suggested of using the natural oxides and carbonates in the ore itself as the precipitant of the ferric sulfate; in other words, the precipitation of the principal impurities in the solutions upon the charge itself. Early tests made on a small scale were very promising, but tests carried out later on a larger scale failed to give the desired results. For the first 15 or 20 days, the copper in the newest charge of ore was sufficient to precipitate all the ferric iron that was contained in the solution passing through the ore. However, as the acid concentration on each charge increased, the precipitated ferric iron was redissolved and eventually accumulated to such an extent that the iron in the solution was in excess of the copper available as a precipitant.

*Use of Sulfur Dioxide*

It was now decided to resort to SO<sub>2</sub> reduction. The general opinion was that this was both unsatisfactory and difficult. This proved to be the case in solutions decidedly acid, but in the case where neutral or slightly acid solutions were used reduction proved quite easy. For these

tests elemental sulfur was used, as it was believed that a gas with the maximum percentage of  $\text{SO}_2$  was most essential and that a rich gas could only be produced by burning elemental sulfur. Owing to lack of knowledge of the operation of sulfur burners considerable trouble was experienced from volatilizing sulfur.

In the sulfite-pulp industry large quantities of sulfur gas are produced and absorbed, and accordingly the method of producing and absorbing the gas used in that industry was investigated. It was found that similar trouble from volatilized sulfur was experienced when making a gas containing above 12 per cent. of sulfur dioxide from elemental sulfur. It was also found that at some plants pyrites were used and that under proper conditions a gas of 12 per cent.  $\text{SO}_2$  could be produced. The only objection in the sulfite-pulp industry to the use of pyrites was the tendency of small calcined particles to be carried into the solution and thence into the pulp. Upon investigation it was decided to make use of the cheap and abundant supply of the high-sulfur low-copper ores of the Bisbee district.

The ore, some 75 T. per day, is unloaded from a trestle on a stock pile, underneath which there is a tunnel provided with a 24-in. (60.9 cm.) belt conveyor. The conveyor delivers the ore to a 13 by 20-in. (33 by 50.4 cm.) Blake jaw crusher, thence to a vertical-shaft 48-in. Symons disk crusher, giving a final product of about 3 mesh. A bucket elevator and a 20-in. conveyor equipped with automatic tripper delivers this crushed ore to eight hoppers situated above four Wedge roasters. These roasters are 22 ft. 6 in. in diameter, have seven hearths, and are belt-driven by  $7\frac{1}{2}$ -hp. motors.

The gas leaving the roasters passes through a short balloon flue to a Cottrell precipitator, where the gas is cleaned of dust before it enters the spray or cooling chamber. The precipitator has forty-eight 13-in. collector tubes and a 65,000-volt circuit is used. At the beginning of operations, the gas cooled to such an extent before reaching the precipitator that sulfuric acid vapor present was condensed and prevented efficient precipitation. Insulating both the balloon flue and the precipitator with magnesia covering and cement remedied this condition. The gas, now practically clean, enters a large sheet-lead chamber locally called the cooling chamber. This chamber is 14 ft. (4.2 m.) square, 94 ft. (28.6 m.) long, and is built of 8 lb. (3.6 kg.) lead supported on a wooden framework. There are 38 nozzles distributed over the top and sides through which "neutral advance" is sprayed to cool the gas before it enters the towers. Between 90 and 100 gal. (340 and 378 l.) of solution per min. are required to supply these sprays. The ferric iron in the solution used in cooling the gas is practically all reduced and the solution joins that coming from the towers. The temperature of the gas in its passage through the spray chamber is reduced from 600 to 150° F. A flue, 20 ft. (6 m.) long and 6 ft.



(1.8 m.) square, connects the spray chamber with the bottom of the first pair of towers, dividing the gas equally between them.

There are six towers arranged in pairs. Two pairs of the towers are part of the original equipment and are 40 ft. (12 m.) high and 20 ft. in diameter. These are built of sheet lead, supported on a steel framework. The top 10 ft. is made of 8-lb. lead, the second 10 ft. of 10-lb. lead, and the bottom 20 ft. of 12-lb. lead. The other pair, built during February, 1918, are 28 ft. (8.5 m.) in diameter, 40 ft. (12 m.) high, and are built of ordinary redwood tank construction, hooped together with iron rods. As an additional precaution against gas leakage, the wooden towers are painted with asphalt and covered with three-ply roofing paper under the hoops. The towers rest upon a reinforced concrete base, provided with a lead pan. All towers are filled with boards  $\frac{3}{8}$  in. (9.5 mm.) thick, 11 in. (32.9 cm.) wide, and placed on edge, the width of a board apart, and in layers. Each layer is laid at right angles to the one immediately below it. The solution is distributed over the top of the towers by a system of launders provided with gas seals. Between the second and third pair of towers is a 60-in. (152 cm.) lead fan. This fan draws the gas from the roasters through the Cottrell precipitator, spray chamber, and third set of towers, and forces it through the second and first sets to the atmosphere. The temperature of the escaping gas is that of the atmosphere.

The solution (or neutral advance) to be reduced travels counter-current to the flow of gas, that is, the most reduced solution comes in contact with the strongest gas. The solution coming from the newest tank of ore is pumped to the top of the third pair of towers by a 9-in. (21.8 cm.) 1600-gal. (6056 l.) per min. centrifugal pump. These are of the Antisell type and work against a 70-ft. head. The solution distributed by launders and gas seals flows down over the filling, thus coming into intimate contact with the rising gas. At the bottom of each pair of towers there is a concrete lead-lined sump, 6 ft. deep and 50 ft. long, into which the solution flows and is then pumped through the next pair of towers. From the first pair of towers the solution is pumped to the second pair, then to the third pair, and then to the so-called settling tank whence it goes to the tank house. The purpose of this settling tank is two-fold: one, to settle out the slime; the other, to get the benefit of the additional reduction that was found to take place in a neutral or slightly acid solution on standing. That the reduction of ferric iron in solution continues for some time after leaving the towers was first pointed out by G. D. Van Arsdale, who observed it during his work with  $\text{SO}_2$  gas as a reducing agent.

A summary of the reduction data for the first year of operation and for March, 1918, is given in Table 15. The successful operation of the electrolytic plant depends, to a large extent, on the operation of the

TABLE 15.—*Reduction Data for First Year and for March, 1918*

	1917-1918	MARCH, 1918
Average tons of sulfide ore roasted per day...	68.5	75.6
Average per cent. sulfur in ore.....	42.7	42.6
Average per cent. sulfur in calcines.....	7.1	6.0
Average roasters in operation .....	3.6	4.0
Average per cent. ferric iron in solution entering towers .....	1.06	0.79
Average per cent. ferric iron in solution leaving towers .....	0.46	0.17
Average per cent. ferric iron in solution entering tank house .....	0.45	0.10
Average per cent. ferric iron reduced .....	0.61	0.69
Average per cent. of total iron reduced.....	57.8	87.3
Average circulation through towers, gal. per min. ....	1005	1324
Average specific gravity of solution through towers .....	1.344	1.305
Average per cent. total iron in solution through towers .....	2.36	2.40
Average per cent. sulfur dioxide by volume in gas entering towers .....	8.1	9.9
Average percent. sulfur dioxide by volume in gas leaving towers .....	1.9	0.8
Pounds of sulfur consumed per pound of ferric iron reduced .....	0.57	0.40
Average tons of new acid produced per day (estimated) .....	38.3	66.3
Average free acid in solution entering towers...	0.37	0.48
Average free acid in solution leaving towers....	1.54	1.70

reduction plant. The reduction plant should be so designed that it will be capable of reducing more ferric iron than will be oxidized during the deposition in the tank house of the maximum quantity of copper. The less ferric iron there is in the electrolyte the higher will be the current efficiency. From the data obtained during the experimental work, only 50 per cent of the theoretical tank-house oxidation took place. Based on these calculations, only four reduction towers were built. After the plant had been in operation a while it was found that the oxidation in the tank house was more nearly 70 per cent. of the theoretical and that four towers were insufficient to meet the conditions that would be imposed upon them by maximum tank-house capacity. Two more towers with a combined capacity of those already in use were then constructed.

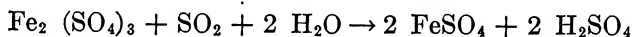
To show the relative reduction in the various towers, an average was made of the solution analyses in and out of each tower for a period extending over one month; see Table 16. These results show the highest reduction taking place in the solution coming in contact with the gas

containing the lowest per cent. of  $\text{SO}_2$ . This is partly due to the facts that the solution entering the first set of towers contains the lowest per cent. of free acid and the highest ferric iron content, and that these towers have the largest capacity. It is evident that the lower the density of the solution, the better will be the absorption and subsequent reduction. The lowering of the density is limited, however, by the operation of the remainder of the process; but it is believed that this fact should be considered as an important point in the operation of the towers.

TABLE 16.—*Analyses of Solution In and Out of Reduction Towers*

	Ferrous Iron, Per Cent.	Ferric Iron, Per Cent.	Total Reduction, Per Cent.
Neutral advance entering towers.....	1.61	0.79	
Solution leaving first pair of towers.....	1.95	0.45	49.5
Solution leaving second pair of towers.....	2.14	0.26	27.5
Solution leaving third pair of towers.....	2.25	0.15	16.0
Solution leaving settling tank.....	2.30	0.10	7.0

It is of interest to note that during the first year 33 per cent. of the total acid required in dissolving the copper content of the ore is produced in the reduction towers according to the reaction:



This, however, represents an increase of only 1.2 per cent. free sulfuric acid in the solution through the towers.

#### ELECTROLYTIC DEPOSITION

The electrolytic tanks are housed in a structural steel building, 166 ft. (50 m.) wide and 280 ft. (85 m.) long, having sides only partly enclosed to give good ventilation. The operating floor is about 15 in. below the top of the electrolytic tanks. The floor in the aisles is of wood grating with the exception of the reinforced-concrete center aisle. The floors at the ends of the building are planks placed at the same level as those of the aisles. The electrolytic tanks are all on the same level, none in cascade.

The cellar, which is open on all sides, had an asphalt floor draining to gutters that lead to a sump at each end of the building. There is 8 ft. of headroom throughout the cellar to permit regular inspection of tanks, piping, and feed-wires.

The electrolytic tanks are arranged in banks with aisles between. There are 12 banks of 10 tanks each and 4 banks of 8 tanks each, making a total of 152 tanks. Each tank is separated from the adjacent tank by

a 3-in. (76.2 mm.) air space. All tanks are made of Oregon pine, lined with 7 lb. (3 kg.) chemical lead. The inside dimensions of the tanks are 29 ft. 7 in. (9 m.) long, 4 ft. 9 in. (1.4 m.) wide, and 4 ft. 3 in. (1.3 m.) deep. These tanks are supported on concrete columns, and are insulated by tile blocks covered with sheet-lead caps. Each tank is provided with a 4-in. clean-out plug. There are also two perforated lead diaphragms, one at each end of the tanks, to assure a uniform circulation. The inlet to each tank is fitted with a 3½-in. diaphragm valve and a 3½-in. glazed stoneware goose-neck for insulating purposes. At the outlet end there is a lead overflow pan fitted with a 4-in. tile pipe suspended in a 10-in. lead boot connected to the discharge pipe.

### *Lead Anodes*

Each tank has 84 anodes, making a total of 12,768 in the tank house. The anodes are of lead containing 3.5 per cent. antimony. The average weight of a lead anode is 215 lb. (97 kg.). They are 40 by 51 by ¼ in. (101.6 by 129.5 cm. by 6.3 mm.) thick, and are suspended by two ¼ by 1¼-in. copper bars secured to the tops of the anodes. The submerged surface of all anodes is 41 by 41 in. The spacing of anodes is 4½ ft. on centers. The distance from the bottom of an anode to the bottom of the tank is 8 in., while that of the cathode is 7 in. Short circuits are prevented to some extent by providing the anodes with eight conical glazed porcelain insulators distributed over the anode faces.

Much doubt was expressed about the life of the lead anode and some very positive statements were made regarding their probable length of life. Continuous service extending for over 1 year has failed to show appreciable oxidation. To obtain definite figures on the disintegration of lead anodes operating under such conditions as these, nine new anodes were carefully marked, weighed, and put into operation. These have been taken out at various times, weighed, and examined. During the first period of operation of 70 days, these anodes showed an increase in weight, due to oxidation, of about 0.94 lb. (0.42 kg.) per anode, or about 0.44 per cent. of the total weight. This amounted to 36 lb. (16 kg.) of lead per ton of copper deposited. During the second period of 42 days, there was an apparent loss in weight of lead; while during the third period, there was no change in weight within the limit of error in weighing. Basing calculations on these figures, it is evident that the oxidation of anodes will not be an important factor.

Previous to our 40-ton tests, little appeared to be known regarding the action of chlorine on lead anodes and much doubt was expressed as to the effect of small amounts of chlorine in a sulfate electrolyte. The water that was used in the 40-ton plant contained only about one-third of the amount of chlorine that is present in the water used in the 5000-ton

plant. Something over 183,000 lb. of electrolytic copper analyzing 99.73 per cent. copper and 0.042 per cent. chlorine were produced in the 40-ton plant, and when checking back on the chlorine introduced into the system, it appeared that about 65 per cent. of it was deposited with the copper. Such also proved to be the case in the 5000-ton plant. The water used for all purposes at the plant averages 0.015 per cent. total chlorine. When starting up the plant and making up leach solution, the chlorine content of the solution gradually increased from 0.015 to 0.021 per cent. when the electrolytic tank house started operations, then decreased until it reached about 0.010 per cent. where it appears to be constant.

### *Cathodes and Starting Sheets*

There are 77 cathodes to a tank, or 9779 cathodes in the tank house, exclusive of starting-sheet blanks. The cathodes are 42 in. (106 cm.) square and are totally submerged. They are suspended upon  $\frac{1}{2}$  by  $1\frac{1}{4}$ -in. (12.7 by 31.7 mm.) copper bars by loops made from starting sheets. The original starting sheets weighed about 15 to 18 lb. (6 to 8 kg.) while the finished cathodes weigh 130 to 140 lb. (58 to 63 kg.). At the present time 127 tanks are used for making cathodes and about 14 to 16 days are required to produce cathodes of the desired weight.

Crane service is provided by two 80-ft. (24 m.) span 5-ton Shaw cranes, operating the length of the building, each serving one-half of the electrolytic tanks. One section of 11 cathodes is removed at a time and carried to the center aisle, where they are washed with hot water to remove the salts and soluble copper. They are then landed on an iron frame to facilitate the hand trucking to the freight cars. Each car is sampled by drilling every twentieth cathode in the center and in diagonally opposite corners. All electrolytic copper, whether cathode or scrap, is shipped to the Raritan Copper Works at Perth Amboy, N. J., where it is melted, brought up to pitch, and cast into commercial shapes.

The cathodes produced have varied from 99.15 to 99.85 per cent. in copper content, the impurities being principally slimes, held by mechanical entanglement. The greater the density of the electrolyte, the lower is the copper content in the cathodes and the greater the insoluble matter, iron, and alumina. The cathodes always contain more or less chlorine, varying from 0.05 to 0.35 per cent. There being no arsenic or antimony in the ore, and very little in the acid, the average arsenic content of the anodes is less than 0.0015 and the antimony less than 0.0005 per cent.

There are 25 tanks operating on starting sheets, each tank containing 77 starting blanks, or a total of 1925 blanks. These are located in the eight-tank banks at the north end of the building. The starting blanks are of rolled 3.5 per cent. antimonial lead 53 by 43 by  $\frac{1}{4}$ -in. (134.6 by

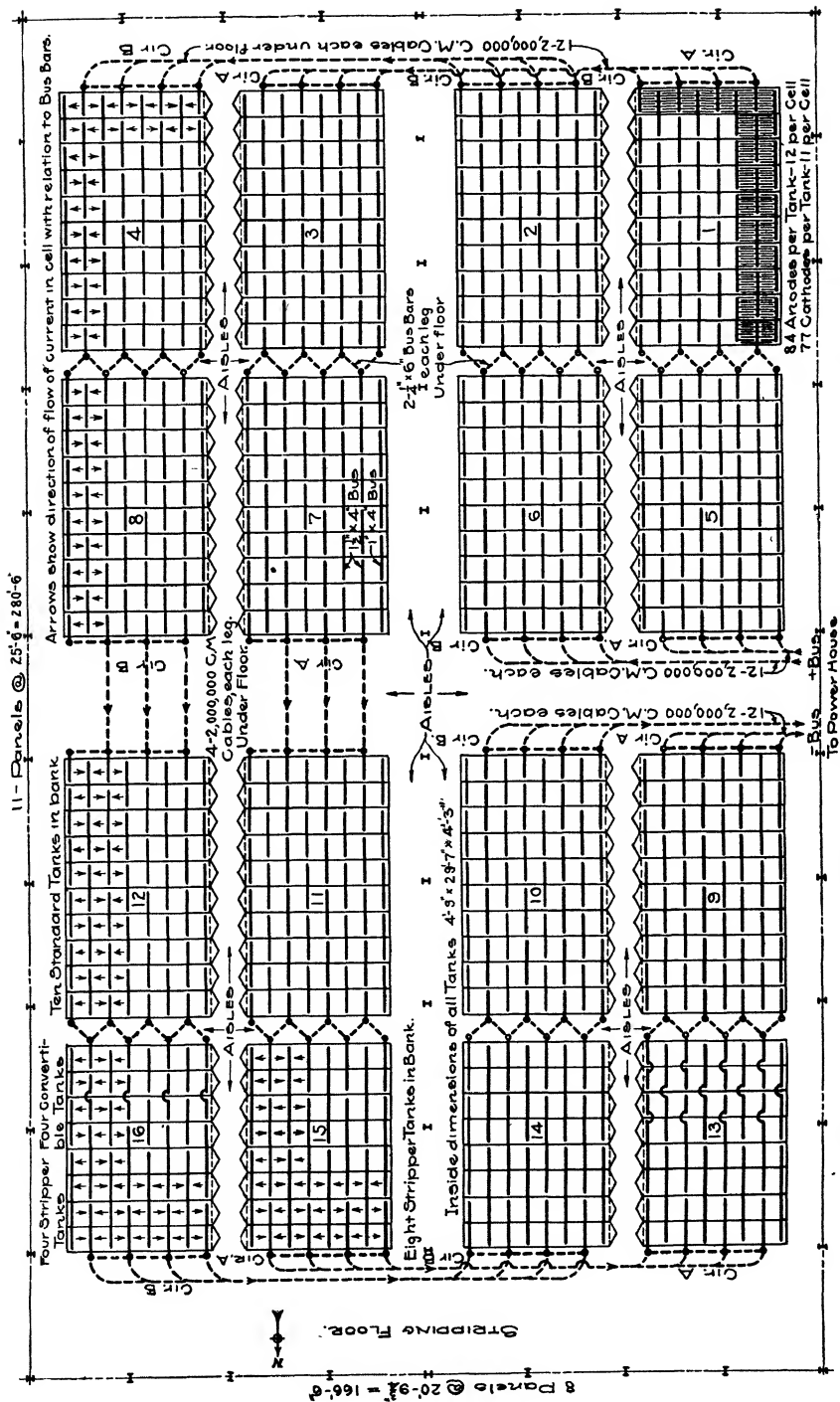


FIG. 6.—FLOW SHEET OF ELECTROLYTIC CONNECTIONS OF ELECTROLYTIC TANK HOUSE.

109 cm. by 6.3 mm.) and are large enough to allow a small amount of trimming, which is done with a squaring shear. Grooved redwood sticks are used at the edge and bottom of the blank to cut the sheet to facilitate stripping, and are found to be satisfactory. The anodes in these tanks are 3.5 per cent. antimonial lead, and are 41 by 52 by  $\frac{1}{4}$  inches. They do not have porcelain insulators, as these tend to spot the starting sheets. The spacing of anodes in these tanks is the same as in the commercial tanks. The tank construction and other details are likewise similar.

Eleven blanks are handled at one time by the crane, and placed on an iron stripping rack provided with a crawl so that the blanks can be carried, one at a time, to the center of the rack, where the starting sheets are removed by two strippers, one stripping from each side. After stripping, the blanks are oiled and placed on the opposite end of the rack to be returned by the crane to the tanks. Four men will strip, under favorable conditions, 924 sheets in 5 hr. The starting sheets, after being stripped and trimmed, are looped on a standard Morrow machine.

The electrodes hang parallel to the flow of solution (or parallel to the length of the tanks) to give a free circulation of the electrolyte. This method of hanging the electrodes was first brought to our attention by the work done at the Butte and Duluth leaching plant.

### *Electric Arrangement*

Alternate busbars extending across the tanks connect the electrodes in parallel and the tanks in series (see illustration). These busbars, placed across the tank, divide it into seven sections or cells. The intermediate busbars are  $1\frac{1}{2}$  in. (38 mm.) wide and 4 in. (101.6 mm.) deep, while the end busbars are 1 in. wide and 4 in. deep. Soldered along the top of each busbar is a triangular piece of copper,  $\frac{1}{2}$  in. high, giving a point contact to the electrode bars. Small maple blocks impregnated with linseed oil insulate cathodes and anodes from opposite busbars. These busbars are supported on insulated iron castings, which in turn rest on the tank cleats. The current for the deposition of the copper is supplied to the tank house by two identical 15,000-amp. circuits, each circuit having 76 tanks in series. This arrangement gives the maximum current density of 8 amp. per sq. ft. of cathode surface when operating under normal conditions. With an average current efficiency of 80 per cent. this means a daily gain of about 10.25 lb. (4.6 kg.) per cathode, or a total capacity of 120,000 lb. of electrolytic copper per day. The drop of potential between anodes and cathodes has averaged very close to 2.00 volts. There is a tendency for the voltage to drop during the summer due to increase in temperature of the electrolyte.

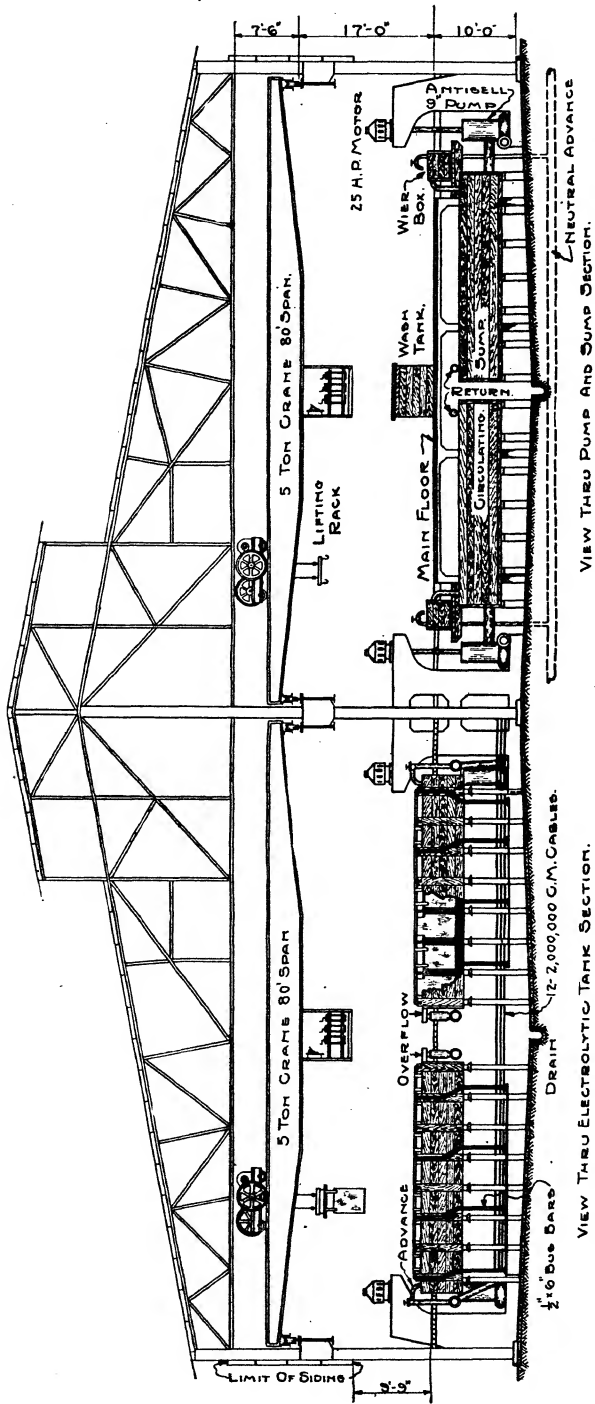


FIG. 7.—EAST AND WEST SECTION THROUGH TANK HOUSE.



*Electrolyte*

The solution flow in the tank house is part of a closed circuit with the leaching and reduction plant, receiving a continuous flow of solution from them. This flow, coming always off the newest ore, then through the towers and settler, is regulated by means of weirs and has varied from 800 to 1500 gal. (3028 to 5677 l.) per min., depending on operating conditions. This volume is divided among the 16 banks of tanks, those on starting sheets getting generally a little more than those on cathodes. By this arrangement each bank of tanks on cathodes receives between 60 and 70 gal. per min. of reduced solution. Each bank unit consists of either 8 or 10 tanks, a sump, and a 9-in. (22.8-cm.) vertical type centrifugal pump of 1600 gal. per min. capacity. Each bank has an individual circulation of 1600 gal. per min. between it and the sump, while an overflow arrangement provides for the return of such a portion of the electrolyte as is equivalent to reduced solution added. Daily analyses are made of the solution entering and leaving the tank house. Since the operating conditions for the month of March were more nearly uniform for the plant as a whole, the analyses of the solution for this period is given in Table 17. The specific gravity of the solution to the tank house (neutral advance) is 1.310 and of the solution from the tank house (acid advance) is 1.305.

TABLE 17.—*Analyses of Solution Entering and Leaving Tank House*

	Solution to Tank House Neutral Advance, Per Cent.	Solution from Tank House Acid Advance, Per Cent.		Solution to Tank House Neutral Advance, Per Cent.	Solution from Tank House Acid Advance, Per Cent.
Cu.....	2.985	2.513	MnO. ....	0.040	0.040
Fe (ferrous)....	2.315	1.660	CaO.....	0.060	0.062
Fe (ferric).....	0.085	0.745	P <sub>2</sub> O <sub>5</sub> .....	0.130	0.130
Fe (total) .....	2.400	2.405	Cl .....	0.0123	0.0110
Al <sub>2</sub> O <sub>3</sub> .....	2.470	2.465	H <sub>2</sub> SO <sub>4</sub> , free....	1.70	2.10
MgO.....	1.360	1.360			

The current efficiency depends on the quantity of ferric sulfate present, due to the reaction between ferric sulfate and metallic copper. The ferric iron content in the solution is kept as low as possible and the conditions shown are as good as can be expected. No doubt with a smaller quantity of total iron present in the solution less would be oxidized, and it was recommended that the total iron be kept below 2 per cent. With the total iron not over 2 per cent., the ferric iron in the elec-

trolyte will probably not exceed 0.5 per cent., the current efficiency will be increased, and more acid will be regenerated.

At the beginning of operations in the tank house, a great deal of difficulty was encountered by the dropping of cathodes in the electrolytic tanks, due principally to the corrosive action of the ferric sulfate on the loops at the solution level, and on that part of the cathode covered by the ends of the loops. Corrosion at the solution line was easily remedied by raising and lowering the solution level in the electrolytic tanks, but the corrosion of the cathode sheet between the loop ends was far more difficult to overcome. Later this condition became worse with the increase in the ferric iron and higher temperature of the electrolyte. The dropping of cathodes not only caused bad short circuits in the tanks but made it necessary, when pulling cathodes for shipment, to pull individual sheets with tongs, which made it almost impossible to handle the daily output of cathodes. Considerable damage was also done to the lead lining of the tanks and the danger from accidents was more than usual. Numerous schemes to overcome this difficulty were suggested and tried, until it was found that by splitting the ends of the loop and attaching them with a Morrow machine in such a manner that the portion of the starting sheet adjacent to the loop was exposed to the deposition of copper, not only the loop, but also the sheet built up, making a good firm joint. Since the adoption of this method no further trouble has been experienced with dropping sheets. Patents have been applied for and allowed covering this improved loop.

The average weight of copper per kw. hr., gross a. c., for the first year was 0.70 lb. (0.3 kg.). It is expected that it will be increased to 0.80 lb. for the coming year.

Since the oxidation of the ferrous sulfate diverts oxygen from the formation of acid, the actual acid regenerated is only about 65 to 70 per cent. of the theoretical. This again shows the importance of keeping down the oxidation to the minimum. This could be done by increasing the flow of solution through the tank house to the permissible limit, or by reducing the quantity of total iron in the solution.

It was originally recommended that the electrolyte be filtered before entering the tank house, with the idea that a purer electrolyte would give better cathodes and starting sheets; this was not done. During the first 6 or 7 mo. the solution coming off the ore went directly to and through the tower into the tank house. As the density of the solution increased more slime was carried from the leaching tanks to the tank house and the quality of the copper produced deteriorated. During November it was decided to settle the solution and one of the leaching tanks was converted for this purpose. This settling, crude as it is, produced a notable difference in both the chemical composition and the physical character of the copper.

Some trouble was experienced in the tank house from anode gases. This was remedied, in the usual way, by keeping a small quantity of oil on top of the electrolyte.

When there was practically complete reduction of the ferric iron in the solution entering the tank house, annoyance was experienced by sulfur dioxide being given off from the solution as it entered the tank house. During this time the free acid in the electrolyte was kept at 3 per cent. and was added prior to the entrance of the solution to the tank house. On adding the acid to the return solution from the tank house this trouble practically ceased. The tank house was started on June 1, 1917, and has been in continuous operation since that time. On June 18, the first electrolytic copper was shipped.

A summary of the tank-house data for March, 1918, is shown in Table 18.

TABLE 18.—*Tank-house Operation, March, 1918*

Total pounds of electrolytic copper produced.....	3,152,800
Total pounds of electrolytic copper shipped.....	3,134,500
Per cent. of total copper shipped as scrap.....	3.9
Average weight per cathode, pounds.....	130
Total gross kw. hr., a. c., charged to tank house.....	4,141,763
Average pounds of copper produced per gross kw. hr., a. c.....	0.76
Average temperature of air in tank house.....	61.5° F.
Average temperature of solution entering tank house.....	86.5° F.
Average temperature of solution leaving tank house.....	93.4° F.
Average current density, amperes per square foot of cathode.....	7.56
Average drop between anode and cathode, volts.....	2.08
Average advance through tank house, gallons per minute.....	1300
Average number of tanks on cathode copper.....	127
Average number of tanks on starting sheets.....	25
Average number of tanks to discard.....	20
Average per cent. copper in solution entering tank house.....	2.99
Average per cent. copper solution leaving tank house.....	2.52
Average per cent. ferric iron in solution entering tank house.....	0.09
Average per cent. ferric iron in solution leaving tank house.....	0.75
Average per cent. total iron in electrolyte.....	2.40
Average per cent. free acid in solution entering tank house.....	1.7
Average per cent. free acid in solution leaving tank house.....	2.1
Average specific gravity of solution entering tank house.....	1.310
Average specific gravity of solution leaving tank house.....	1.305

Adjoining the tank house on the west is the power house. This was designed by C. C. Moore & Co., of San Francisco, and is a structural steel building 200 ft. (60 m.) long, 126 ft. (38 m.) wide. Five 822.6-hp. Sterling boilers equipped with economizers and automatic fuel-oil control generate the steam. The steam, at 240 lb. (108 kg.) pressure and a total steam temperature of 520°, is delivered to one of two

turbo-generators with a normal rating of 7500 kw. each. Either of these generators is capable of handling the entire plant load. Each exhausts into a Wheeler surface-type condenser. The circulating water used for the condensers is cooled by means of a  $150 \times 400$ -ft. spray pond. By drawing water for the leaching plant from this spray pond, the salts and incrusting solids are prevented from accumulating.

All power generated is delivered to the station buses, which are enclosed in a concrete structure. Leading from these buses are ten 2300-volt feeders, the three largest carrying power to three Westinghouse motor-generator sets, each consisting of a 2400-hp. synchronous motor, direct-connected to two 170-volts, 5000-amp. d. c. generators, which furnish the current for the tank house, while the seven smaller feeders supply a. c. current which is used by the crushing, leaching, and reduction plant, well, mine, and townsite. About 600 bbl. of oil are consumed by the plant per day. This is supplied through a 6-in. oil line from two steel tanks 60 ft. in diameter by 30 ft. high, each with a capacity of about 15,000 barrels. The average kw. hr. per barrel of oil for the year has been 314.2. The oil used at the plant will average 18,766 B.t.u. per pound of oil.

#### DISCARD FOR PURIFICATION OF ELECTROLYTE

As previously mentioned, only about 45 to 50 per cent. of the total acid used in an 8-day leach is utilized in dissolving copper. The remainder is used in dissolving impurities. If copper only is removed from the solution, the other substances will gradually accumulate and the solution will reach a condition where it will become sluggish in dissolving the copper from the ore. To keep the solution active, it is evident that a portion must be discarded and replaced with fresh water. The quantity of solution discarded per day must contain impurities equivalent to the amount dissolved per day, if the accumulation is to be avoided. In the experimental work it was found that, under similar conditions, nearly all the substances that went into the solution were present in a fairly constant ratio to one another. Of the various impurities dissolved, iron is the most easily and quickly determined and was used as the indication of the quantity of solution necessary to be discarded. Our experimental work clearly demonstrated that the best results are obtained when the total iron in the solution does not exceed 2 per cent. and the specific gravity does not exceed 1.28. To maintain the most efficient conditions, 90 gal. (340 l.) per min. discard was reported as necessary. This was later proved to be correct for conditions in the 5000-ton plant, as shown by the summary in Table 19.

The most economical solution to discard is that containing the greatest quantity of impurity and the least quantity of free acid per unit

of copper. For the first 7 months of operation, neutral advance was the most desirable to discard. This solution averaged 3 per cent. copper, 0.2 to 0.5 per cent. free  $H_2SO_4$ , and 2.4 per cent. total iron, of which 0.6 to 0.8 per cent. is present as ferric iron. An attempt was made to precipitate the copper from this solution on heavy scrap, but the action

TABLE 19.—*Discard Solutions*

	Gallons per Minute for 24 Hours	Specific Gravity of Solution	Total Iron in Solution, Per Cent.	Average, Days, Leach- ing Period	Number of Charges Excavated
May, 1917.....	none	1.15	0.34	9.3	3
June, 1917.....	none	1.24	1.60	16.1	14
July, 1917.....	21	1.35	2.36	13.9	18
August, 1917.....	50	1.38	2.60	12.0	17
September, 1917.....	90	1.38	2.56	10.5	23
October, 1917.....	83	1.36	2.44	9.9	23
November, 1917.....	80	1.34	2.36	9.5	23
December, 1917.....	70	1.33	2.38	9.3	25
January, 1918.....	72	1.32	2.26	7.8	28
February, 1918.....	73	1.31	2.39	9.3	24
March, 1918.....	75	1.31	2.40	7.2	32
April, 1918.....	82	1.32	2.49	7.3	29

was very sluggish. In addition to this difficulty, the launder space designed to treat 30 gal. (113 l.) per min. proved to be insufficient. As the cost of producing cement copper is about 2 c. more per pound than that of electrolytic, it was of prime importance to reduce this amount to

TABLE 20.—*Solutions going to Iron Precipitation Launderers*

	Copper, Per Cent		$H_2SO_4$ , Per Cent.		Ferric Iron, Per Cent.		Average Current Efficiency, Per Cent.
	In	Out	In	Out	In	Out	
First bank.....	3.14	2.72	0.95	1.64	0.45	1.06	73.0
Second bank.....	2.72	2.30	1.64	1.93	1.06	1.51	55.0
Third bank.....	2.30	2.06	1.93	2.23	1.51	1.71	46.0
Fourth bank.....	2.06	1.71	2.23	2.62	1.71	1.82	47.0
All banks.....	3.14	1.71	0.95	2.62	0.45	1.82	

the minimum. Since when using neutral solution for discard nearly one-third of the total copper production would be produced as cement copper, an investigation was made to produce a solution containing the greatest amount of impurities per unit of copper. During December,

four electrolytic banks of 10 tanks each were connected in series and the effluent from the last bank of tanks was sent to the iron precipitation launders. The average analyses of the solution through each bank of tanks, with a flow of 73 gal. per min., was as shown in Table 20.

The results will vary with the amount of solution flowing through the tanks. The action of the effluent from the last bank of tanks was anything but sluggish and the precipitation was rapid. However, the high iron consumption and the low power efficiency made this arrangement doubtful economy and two of these banks were put on regular tank-house practice. This discard solution, containing a certain amount of free acid, precipitates copper more rapidly than a neutral solution. The cement copper so produced is easily washed from the scrap iron. This method of discarding solution produces a smaller amount of cement and a greater amount of electrolytic copper.

#### RECOVERY OF COPPER FROM DISCARDED SOLUTION

The original cementing equipment consisted of six sections of reinforced-concrete launders. These are arranged in two parallel rows of three sections each. They are each 60 ft. (18 m.) long, 10 ft. (3 m.) wide, and 4 ft. 6 in. (1.37 m.) deep, giving a total launder capacity of 16,200 cu. ft. (458 cu. m.) or 121,500 gal. (459,857 l.). The bottom of each launder slopes toward three side cleanout gates. The scrap iron rests upon a grated wood floor. The solution flows through each one successively, but may be by-passed to allow the cleansing and refilling of any of the sections. From these launders the waste solution was to go to the desert.

The launders provided to handle the solution were found to be insufficient and the scrap iron was too coarse for good work. Only about 65 per cent. of the copper in the solution was precipitated during the first 3 mos., and the effluent from these launders was impounded in large ponds and later pumped back to the new launders. The construction of eight additional temporary wooden launders was begun, lighter scrap iron was bought, and the copper content of the waste solution was thereby reduced. Of the eight temporary wooden launders, one is 14 ft. (4 m.) wide, 280 ft. (85 m.) long, and  $2\frac{1}{2}$  ft. (0.75 m.) deep, and seven are 10 ft. (3 m.) wide, 280 ft. long, and  $3\frac{1}{2}$  ft. (1.05 m.) deep. These have a total capacity of 78,400 cu. ft. (2218 cu. m.), or 588,000 gal. (2,225, 580 l.), thus giving a total capacity of both concrete and wooden launders of 94,600 cu. ft. (2677 cu. m.) or 709,500 gal. (2,685,457 l.). The wooden launders are connected with the concrete launders by a 6-in. lead pipe line.

Under good operating conditions about half the copper is precipitated in each set of launders. When a launder is no longer efficient, the solu-

tion to that launder is by-passed and the solution remaining in it is drained. The cement copper in the concrete launders is shoveled out onto wooden platforms, where the iron is hand-picked, and the copper is loaded into box cars for shipment to the smelter.

The cement copper, being practically saturated with ferrous and aluminum sulfates, dries out very slowly. Recently a concrete sump 20 by 75 by 3 ft. (6 by 22 by 0.9 m.) was constructed near the wooden launders, into which the cement copper can be flushed and washed. A locomotive crane removes the copper in a small bucket to shallow wooden bins, where it is dried for shipment. This part of the process has proved to be a problem and methods are being developed to handle the material more economically.

The solutions entering and leaving these launders during an average month are as follows:

	H <sub>2</sub> SO <sub>4</sub> , Per Cent.	Copper, Per Cent.	Ferrous Iron, Per Cent.	Ferric Iron, Per Cent.	Specific Gravity
Solution going to concrete launders.....	2.34	2.12	1.07	1.44	1.31
Solution coming out of concrete and going into wood launders.....	0.77	0.63	4.51	0.13	1.31
Solution from wood launders to desert...	0.08	0.02	4.54	0.08	1.25

### *Metallizing Tests*

Anticipating the difficulty that will probably be experienced in obtaining suitable scrap iron for precipitation purposes, the company decided to investigate again the plan, first suggested by Mr. Croasdale in 1912, of reducing the iron oxide in calcine to sponge iron. There was no doubt but that this material could be produced and all efforts were directed to devise a furnace for that particular purpose. However, the experiments were brought to a close before this had been accomplished. About this time the experimental staff of the Anaconda Copper Co., working along similar lines, developed a revolving intermittent metallizing furnace of the Brückner type. The results of these tests have been fully described in the *Transactions*.<sup>3</sup>

When we decided again to investigate metallizing, arrangements were made with the Anaconda company to install one of their Brückner furnaces and to furnish us an experienced operator. These tests are still in progress at this writing. We feel convinced, from the results already obtained, that with a high-iron, low-sulfur calcine, nearly complete metal-

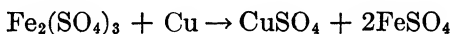
<sup>3</sup> Frederick Laist and F. F. Frick: *Trans.* (1914) 49, 691.

lization of the available iron is possible and with coal and oil at a reasonable price, the cost of sponge iron will compare favorably with the present cost of scrap.

At the present time between 20,000 and 30,000 lb. (9071 to 13,607 kg.) of copper are being precipitated per day, with an iron consumption of about 2 lb. (0.9 kg.) per pound of copper. If calcines (metallized) were to be used the principal saving would be in the cost of handling the precipitate.

### RESOLUTION OF CEMENT COPPER

It was originally intended to make use of the ferric sulfate present in the oxidized tank-house solution as a solvent of the cement copper, thereby increasing the production of electrolytic copper and at the same time avoiding the handling of cement copper. Cement copper, when clean and finely divided, dissolves readily in ferric sulfate with formation of copper and ferrous sulfates:



By so doing not only is the cement copper dissolved, but the ferric iron is reduced also. The plan was to hose off the loosely adherent copper from the scrap iron to the bottom of the tank and then flush it into one or more circular lead-lined tanks, called agitators. These tanks are 20 ft. (6 m.) in diameter and 6 ft. (1.8 m.) deep, provided with a stirring device, driven by a small motor; 125 gal. (473 l.) per min. of tank-house return solution can be circulated through each tank.

This plan of redissolving the cement copper is only carried out when the operating conditions of the plant require it. The principal objections to the continuous use of such a plan are the difficulty of introduction of the copper to the tanks, the solution dilution that it incurs, and the tendency to increase the impurities, which in turn increase the quantity of discard necessary.

The agitators were started on July 1, 1917, and were continuously operated until Sept. 1, 1917, when it was decided to ship all cement copper possible. During February, the grade of the ore dropped for a short time and it was desirable to increase the copper contents of the electrolyte, and the agitators were once more put into commission. Careful comparison of the present cost of shipping and treating this copper and the cost of redissolving give the latter method a small advantage. In other words, the agitators have not given the results that were anticipated. If, however, a method could be devised whereby the copper could be washed, drained, and then transferred without dilution, it would be a more profitable operation.



## SUMMARY

In order that the reasons for some of the present operating conditions may be thoroughly understood, a short account of the events that led to these conditions will be given. Much time having been consumed in the development of the process, the management was eager to begin operations and have this company enter the producing class.

On Apr. 15, the crushers were given a tryout and filling of the leaching tanks was begun. These were filled with water, both before and after charging with ore, to test for leaks. By May 1, enough tanks had been filled to begin leaching and everything was in readiness except the solution and wash-water pumps. The pumps appeared to be out of balance and much trouble was experienced in the correction. However, by May 16, enough had been accomplished to guarantee continuous operation of the plant. On May 16, 300,000 gal. of 3 per cent. sulfuric-acid solution was made up and pumped on the first charge of ore. This solution was circulated through the first charge the first day, through two charges the second day, and so on each day, more solution being made up to keep the acid concentration at 3 per cent. on the oldest tank.

Unfamiliarity with the piping, leaky tanks, inability of the mine regularly to deliver 5000 T. of ore per day, together with the usual initial operating troubles, resulted in the washing and excavation of only three tanks during May. These conditions rapidly improved, so that over 75 per cent. of the required ore was treated during the first year, and nearly 90 per cent. of the required copper produced in the plant. This was due to the fact that the ore mined averaged nearly 0.1 per cent. better than was anticipated. During this time tanks were not excavated until a new one had been charged and put into circuit, which led to the use of an excessive quantity of acid and accomplished little but the fouling of the solution with impurities.

When the solution carried  $2\frac{1}{2}$  per cent. copper, it was advanced to the tank house. By June 1, the electrolytic tanks had been filled and deposition begun. The towers were not started simultaneously with the tank house and by the time of their introduction into the circuit on June 6, the ferric iron had reached 0.7 per cent., the efficiency had dropped, and trouble started.

The first cathodes were drawn on June 15. The solution, clear and of low gravity, produced cathodes analyzing 99.85 per cent. copper. Later as the solution became foul and the density went up to 1.42, the cathodes produced analyzed but 99.1 per cent. copper. The gravity of the solution rose very rapidly and discarding of solution became imperative if the plant was to continue to operate. On July 15, the construction of additional cementing launders was decided upon and as fast as men and material could be obtained these were built and put into opera-

tion. Before these launders could be finished, the conditions reached a point where solution had to be discarded irrespective of copper content. The solution coming from the launders was run in a pond and a portion of the copper content was recovered later by treatment in the launders. The first cement copper produced was dissolved in tank-house return solution. This was abandoned for the time being and on Aug. 20 the first car of cement copper was shipped to the smelter.

The original report on the process to be adopted, among other things, recommended that a cooling chamber be installed at the entrance of the gas to the towers. During August, the efficiency of the towers was falling and it was found that a part of the solution coming in contact with the hot gases was evaporated to crystallization, thus choking the gas entrance to the towers. It was then decided to install a cooling chamber. By September, it was realized that the towers lacked reduction capacity and two more would have to be built; these were finished and put into operation Feb. 16, 1918. The fourth roaster was found necessary when the third pair of towers was started. These towers, the cooling chamber, the additional launders, and the wood lining in the leaching tanks represent the only important changes made.

It is reasonable to suppose that with a solution of 1.25 density the extraction, reduction, and tank-house efficiency would be improved. A lighter solution will be a more active lixiviant, the tailings will be more easily washed, less slime will be carried to the tank house, and better cathodes will be produced. The reduction in pumping and repair costs alone might prove to be quite an item.

In conclusion, while the fundamental reactions are simple the attainment of the desired results has been more difficult. Small causes are apt to bring about big results and, as in case of any purely technical operation, the best results can only be secured by the most careful supervision. Neither metallurgically nor mechanically is there anything radically new in the process except that it has been a commercial success from the start.

## APPENDIX

In order to increase the value of the paper the following data, which is a part of the annual metallurgical statement to Jan. 1, 1919, is appended. To give a complete discussion of the noticeable changes is not possible at this time. This supplement, however, will be concluded with a short discussion of a few of the more important variations.

*Summary of Results*

	1917	1918	To JAN. 1, 1919
Total tons of dry ore charged to leaching plant .....	780,211	1,760,862	2,541,073
Total number of tanks charged .....	156	355	511
Total copper contents, per cent.....	1.685	1.465	1.535
Soluble copper contents, per cent.....	1.632	1.405	1.477
Insoluble copper (probably present as sulfide), per cent .....	0.053	0.060	0.058
Average proportion of ore on 3-mesh screen, per cent .....	27.46	26.75	27.01
Average proportion of ore on 4-mesh screen (cumulative), per cent.....	42.46	43.43	43.13
Average proportion of ore through 20-mesh screen, per cent ..	18.88	18.43	18.63
Total number of tanks excavated .....	146	355	501
Average moisture in tailings, per cent.....	11.02	10.73	10.81
Total copper in tailings, per cent.....	0.371	0.280	0.306
Copper in laboratory washed tailings, per cent .....	0.270	0.227	0.239
Water soluble copper in tailings, per cent.....	0.101	0.053	0.067
Average pounds of water soluble copper per ton of tailings ..	2.02	1.06	1.34
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Average number of days leached.....	10.6	7.59	8.1
Average gallons per minute advance through ore.....	1020	1139	1095
Circulation in tank, gallons per minute .....	4500	4500	4500
Average specific gravity of neutral advance. ....	1.360	1.318	1.333
Average free sulfuric acid going on oldest ore, per cent .....	2.80	2.90	2.86
Average sulfuric acid in solution coming off newest ore, per cent .	0.35	0.42	0.39
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Average gallons per minute through towers .....	955	1139	1071
Total iron in neutral advance to towers, per cent .....	2.34	2.36	2.35
Ferric iron in neutral advance to towers, per cent .....	1.09	0.84	0.93
Ferric iron in neutral advance from towers, per cent .....	0.60	0.21	0.354
Roasters in service.....	3.0	3.9	3.54
Average tons of ore roasted per day.....	64.2	71.7	69.6
Average sulfur contents of ore, per cent. ....	42.9	42.2	42.4
Average sulfur contents of calcines, per cent ..	7.8	7.6	7.7
Average sulfur dioxide in gas to towers, per cent ..	7.63	8.00	7.85
Average sulfur dioxide in gas from towers, per cent ..	2.44	0.82	1.39
Average sulfur consumed per pound of ferric iron reduced ..	0.67	0.46	0.54
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Average gallons per minute through tank house .....	955	1139	1071
Average copper contents of solution entering tank house, per cent.....	2.96	3.12	3.060
Average copper contents of solution leaving tank house, per cent	2.47	2.71	2.621
Average copper contents removed from solution through tank house, per cent.....	0.49	0.41	0.439
Average ferric-iron contents of solution entering tank house, per cent.....	0.60	0.21	0.354
Average ferric-iron contents of solution leaving tank house, per cent.....	1.08	0.82	0.916
Per cent. of theoretical oxidation .....	55.9	85.0	73.1
Average current density, amperes per square foot .....	6.20	7.26	6.91
Average voltage between anode and cathode ..	1.95	1.98	1.97
Average weight per cathode shipped .....	130.7	112.4	117.09
Number of tanks on cathodes .....	116.	125.5	122.3
Number of tanks on starting sheets ..	22.2	26.5	25.1
Number of starting sheets made .....	155,543	365,547	521,090
Starting sheets scrapped, per cent.....	11.50	9.56	10.2
Electrolytic copper shipped, pounds .....	12,661,215	31,745,480	44,406,695
Copper per gross a. c.—kw. hr., pounds .....	0.669	0.697	
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Total acid (60° B. sulfuric acid) charged to plant, tons .....	40,278.60	63,185.08	103,463.68
60° B. acid per ton of ore leached, pounds .....	103.2	71.2	82.6
60° B. acid per pound of copper dissolved, pounds.....	4.03	2.97	3.37
Average per day 60° B., tons .....	175.10	173.06	173.88
Average copper dissolved per ton of ore leached, pounds .....	25.62	23.95	24.44

*Summary of Results—(Continued)*

	1917	1918	To Jan. 1, 1919
Average total copper dissolved, per cent.....	77.85	81.17	80.06
Total copper produced as electrolytic, per cent.....	79.60	77.26	77.93
Total copper produced as cement, per cent.....	20.40	22.74	22.07
Average copper in cathodes, per cent.....	99.47	99.49	99.48
Average copper in cement copper, per cent.....	68.91	54.46	57.23
Average of all solutions going to cementing launders, gallons per minute.....	65.0	131.3	109.0
Average of copper per day in all solutions going to launders, pounds.....	29,563	33,956	28,775
Total copper recovered from solutions to launders, per cent.....	82.05	98.215	93.03
Cement copper shipped as 100 per cent. copper, pounds .....	3,425,907	11,382,487	14,808,394

The foregoing results show better extraction and better washing for 1918. The per cent. of soluble copper in the tailings will be further reduced by the use of a fifth wash water, which was started on Nov. 9, 1918. Operating conditions during this last year resulted in nearly uniform leaching conditions. The local circulation on the ore has been varied from time to time but 4500 gal. per min. appeared to give the most satisfactory condition. During the past 3 mo. the free acid in the solution going on the oldest ore has been 2.65 per cent. and the extraction has been as good as the average. It now appears that it will be possible to leach with a much lower acid concentration than at first thought necessary. The difference between a 2.65 and a 3.00 per cent. free acid means a saving of approximately 50 tons of acid per day. An acid balance covering the past year's operation indicates that nearly 68 per cent. of the total acid neutralized by the ore was used in dissolving impurities, and 32 per cent. was used in dissolving copper. This statement also shows that of the total acid neutralized by the ore 49.1 per cent. is imported acid, 35.6 per cent. is tower acid, and 15.3 per cent. is regenerated tank-house acid. The total 60° B. acid neutralized per ton of ore for 1918 is 148.6 pounds.

During the year a slight warping of the lead anodes was noticed and these were removed from tanks and straightened, resulting in a better tank-house efficiency. This warping was apparently due to oxidation. To keep the solution from fouling it was necessary to send 109.9 gal. per min. of solution to the precipitation launders. Of this solution 84.9 per cent. was from the tank house, 12.0 per cent. was excess wash water, 2.1 per cent. was tower solution, and 1.0 per cent. was tank-house drainage. During the year the larger part of the copper solution that had been impounded in ponds during the previous year was returned to the precipitation launders and the copper recovered.

During the past year, the plant has undergone no radical changes or alterations. All mechanical equipment, including pumps, piping, and lining have proved satisfactory in both design and material. Plans are being prepared for a new cementation plant, consisting of twelve circular precipitation tanks, classifiers, and filters. This plant is to be spanned

by a crane, which will charge the scrap iron to the precipitating tanks. This plant is being designed to handle the copper precipitated mechanically and to thoroughly dry it.

Problems are constantly arising, the solution of which tends to materially simplify operation. These are being investigated in order of their importance and furnish a constant source of interest. The past year has been considered a successful one by all connected with the work and basing our predictions on the experience gained, we predict even better results for the year 1919.

## DISCUSSION

C. A. ROSE, New York, N. Y. (written discussion\*).—Without doubt the excellent results obtained at Ajo will cause surprise among metallurgists; 75 per cent. average capacity and 80 per cent. extraction during the first year of operation of a plant using a new metallurgical process are figures that speak for themselves.

It is obvious that the most troublesome feature of the process is the fouling of solutions. The effects of this are most evident in the electrolytic tank house; but apparently the solutions are so heavily loaded with salts that their dissolving power is also impaired. This is indicated by the fact that copper comes into solution during washing after the leaching of the ore is completed, as shown by the decreasing ratio of acid to copper in the various washes tabulated in Table 4, and also by the fact that 4.9 per cent. of the total copper in the ore remains in the tailings in a soluble form.

Even though the extraction in leaching and the ampere efficiency in electrolysis would be benefited by keeping the salts in solution at a lower concentration, it is doubtful whether it would pay to decrease these salts by discarding more electrolyte without other changes in the process, since this would increase the amount of cement copper produced, which is already very large. If, however, it would not disturb the process otherwise, this object could be accomplished by increasing the range of electrolysis, so that solution going to the tank house would contain, say, 4 per cent. copper and 1 per cent. acid, and the solution returned to the leaching plant  $1\frac{1}{2}$  per cent. copper and 4.8 per cent. acid, and by increasing the volume of solution to be discarded, this to be taken from the electrolyzed solution returned to the leaching plant. By this arrangement, more salts will be removed from the system per ton of cement copper produced; and if sufficient solution is discarded to keep the iron content down to  $1\frac{1}{2}$  per cent. it would be feasible to recover the copper from this wasted solution by electrolysis. In order to keep the iron down to  $1\frac{1}{2}$  per cent., it would be necessary to discard a volume of solution about

twice as great as at present. This would mean an additional loss of 25 to 30 tons of acid per day in the wasted solution, but it is very probable that the increased extraction in the leaching plant and the better ampere efficiency in the tank house would more than compensate for this loss of acid.

The average ampere efficiency in the tank house, calculated from the average voltage and pounds of copper deposited per kw. hr. given in the tabulation, was about 63 per cent. If the iron in the electrolyte is kept below  $1\frac{1}{2}$  per cent., there is no doubt that the ampere efficiency in the commercial tanks can be increased to more than 80 per cent. and that the copper recovered from wasted solution will be deposited out to as low as  $\frac{1}{2}$  gm. per liter, with an average current efficiency of at least 50 per cent. It would also seem advisable to use a higher current density, since this would increase the ratio between the copper deposited to the copper dissolved from the cathodes by the ferric sulfate in the electrolyte.

Apparently 65 per cent. of the oxygen liberated at the anode is taken up by the ferrous sulfate in the electrolyte and yet the drop of potential between the anode and cathode is 2 volts. It would seem, therefore, that very little benefit as a depolarizer is derived from the reduction of the solution by  $\text{SO}_2$ .

The advantage of the great amount of circulation in the leaching tanks is not apparent unless it be aeration of solutions. Also, it is doubtful whether the ferric iron in the solutions has any beneficial effect in the leaching operations, since the solution delivered by the leaching plant contains more ferric iron than that received from the tank house. Perhaps the continued dissolving of copper during washing operations is due to the aeration which results when the ore is drained between washings. If this is the case, it might pay to drain the ore several times during the leaching period. A saving of about 8 per cent. of the power generated could probably be made by substituting rotary converters for motor-generator sets.

STUART CROASDALE,\* Denver, Colo. (written discussion†).—It is very gratifying, on reading the paper of Messrs. Tobelmann and Potter giving the results of the first years' operation of the New Cornelia Copper Co., to learn how closely these results check with those obtained at the pioneer experimental plant for this ore at Douglas, Ariz., where these gentlemen so ably and so faithfully assisted me in blazing the way for the plant now in operation at Ajo. A few comparisons are interesting: 192.4 tons of ore was treated at the Douglas plant, having an average content of 1.43 per cent. copper; the copper left in the tailings was 0.32 per cent. At Ajo 1,345,000 tons of ore was treated during the first year,

\* Consulting Engineer.

† Received Feb. 19, 1919.

having an average content of 1.63 per cent. copper; the copper left in the tailings was 0.338 per cent.

Perhaps the most important part of the paper is that dealing with the consumption of acid and the fouling of the solutions. In my preliminary laboratory experiments at Douglas, I varied the strength of the acid lixiviant from the amount theoretically necessary to dissolve the copper in the ore, up to a 10 per cent. solution. The theoretical amount necessary to dissolve the copper in this case happened to be a 3 per cent. solution of 100 per cent.  $\text{H}_2\text{SO}_4$ . This acid solution, or lixiviant, corresponds to the acid advance mentioned by Tobelmann and Potter. Before precipitating the copper, the excess of acid was neutralized by circulating the lixivium through new ore, as is done at Ajo.

In some of the tests made in the experimental plant at Douglas, the acid in the lixiviant was reduced as low as 1.7 per cent. but, in order to acquire information as quickly as possible, most of the tests were made with a 10 per cent. acid solution and the leaching extended over a period of three days, exclusive of washing. My tests with a 10 per cent. acid solution gave an average consumption of 3.15 lb. of 100 per cent.  $\text{H}_2\text{SO}_4$  per pound of copper dissolved. My laboratory tests with a 5 per cent. acid solution gave an average consumption of 2.5 lb. of acid per pound of copper dissolved; while in the experimental plant, the same strength of solution gave a result of only 2.0 lb. of acid consumed after a period of five to six days leaching, exclusive of washing. In tests made at the 40-ton experimental plant at Ajo, the consumption of acid was 2.8 lb. per lb. of copper dissolved when a 3 per cent. lixiviant was used, and the leaching extended over a period of eight days.

The first year's operation of the large plant shows an average consumption of 2.76 lb. of acid per pound of copper dissolved when using a 3 per cent. acid lixiviant and extending the leaching period to eight days. During the last four months of the year, however, the acid consumption was reduced to 2.14 lb. and the leaching period to six or seven days. Apparently the 3 per cent. acid advance has yielded no less acid consumption than the 5 per cent. solution that I used, while the time of leaching has been necessarily extended as a consequence of the weaker lixiviant.

On comparing the analyses of the lixiviums that I obtained, using both strong and weak acid solutions, with those obtained during the first year's operation at Ajo, the question arises whether the anticipated advantage has been gained in the direction of cleaner lixiviums by using the lowest strength acid lixiviant.

Probably all of the soluble iron in this ore is derived from the oxidized pyrite and chalcopyrite. Owing to the climatic conditions of the Ajo district, the oxidation of these minerals has left both the physical and chemical conditions of the iron in such form as to be quite readily dis-

solved in an acid solution of any strength. The iron seems to follow the copper rather slowly in relative solubility, but it passes into solution with increasing rapidity as the time of leaching is lengthened, even with a weak acid solution.

The soluble alumina is probably derived from the decomposed feldspars that have been kaolinized by ordinary weathering of the rock assisted by the acid from the oxidation of the pyrite. The alumina follows the iron closely in relative solubility. It also passes into solution with increasing rapidity as the time of leaching is lengthened, to a greater degree than the iron.

Of course no leaching is accomplished if the lixiviant is not sufficiently acid to overcome the basicity of the ore and a positive disadvantage results. Copper is necessarily precipitated in the ore, which has to be redissolved by the more acid lixiviants that follow. Basic sulfates of iron and aluminum are almost sure to form, which will have a tendency to separate out as a slimy flocculent precipitate in the neutral or weakly acid lixiviums.

It would be interesting to learn if the so-called slimes that have given trouble in the  $\text{SO}_2$  towers, electrolytic tanks, and scrap-iron launders, are not largely basic sulfates of iron and aluminum rather than colloidal slimes from the ore. If this can be determined, it might be suggestive in the future treatment of the ore. With upward percolation, however, I presume the tendency for the colloidal slimes to follow the solutions is greater than it would be with downward percolation.

I have gone into this subject somewhat at length in order to support my belief and original recommendation in favor of a stronger acid advance and a shorter period of leaching. I do not recommend using the 10 per cent. acid lixiviant that I used at Douglas, because that was unnecessarily high for the reasons already stated; but with a knowledge of the results now at hand, I do believe a 5 per cent. solution can be used advantageously over the 3 per cent. solution now in use without increasing the consumption of acid per pound of copper dissolved.

The next interesting feature of the year's work is the resumed effort to utilize the iron in the calcines, in the form of sponge iron, as a precipitant for copper. As the authors have stated, my investigations along this line at Douglas were brought to a close about the time we had learned nearly everything we should not do and before we had been able to devise a furnace of commercial value that would effect a complete reduction of the calcines at a comparatively low temperature. This work was taken up later by the Anaconda Copper Co. and sponge iron was made in quantity at Anaconda by heating calcines and coal in a Brückner furnace by means of an oil burner. In discussing this subject with Mr. Laist a few years ago, he said they found that the reduction of the iron oxide to  $\text{FeO}$  was accomplished quickly at low temperature;



but to complete the reduction to metallic iron required an unexpectedly high temperature, which added considerably to the expense, and since they always had sufficient scrap iron on hand to meet their precipitation requirements they abandoned the reduction of calcines.

My own investigation, when using this method of reduction, had already confirmed the Anaconda results and had led me to seek lines of less resistance that would yield metallic iron at lower temperatures, which I had good reasons to believe could be obtained. That iron oxide, particularly as it occurs in calcines, can be completely reduced to metallic iron at temperatures under 1600° F., or 900° C., has been demonstrated by myself as well as other investigators. In common parlance, this is a cherry-red heat. There is no difficulty about the chemical part of the process. The problem lies in constructing a commercial furnace that will enable one to bring about the chemical reactions when operating continuously on a large scale. The conditions of the problem are not insolvable.

F. S. SCHIMERKA,\* Clifton, Ariz. (written discussion).—Messrs. Tobelmann and Potter have made a very notable contribution to the hydrometallurgy of copper by revealing with painstaking thoroughness the inner workings of the Ajo plant, the development of which has been followed with great interest by all whose attention is directed toward the wet treatment of oxidized copper ores. We have been treated to a metallurgical feast and, as regards the abundance of data furnished, with a generosity deserving of high appreciation.

It cannot be denied that the animosity against the treatment of copper ores by wet methods has only lately been overcome. Metallurgists have been less outspoken in this animosity than was capital, which hesitated to engage in a large-scale project along new and substantially untried lines. The practicability of the process required demonstration by leaders and it has been furnished by the success that has attended large enterprise. The frank statement of the authors that neither metallurgically nor mechanically is there anything radically new in the process detracts nothing from their achievement, which consists in the surmounting of very considerable difficulties in the way of practical application of known principles.

The two distinct phases of the process are the lixiviation of the copper and the deposition of the metal. Of these the last is, in the larger number of cases, the crux of the process. The reclamation of the metal from its liquors has tried the skill of the metallurgist to no trifling degree. Setting aside the method of precipitation by scrap iron, the brutality of which, borrowing the expression from a leader, has not interfered with its popularity and application as a last resort, the task of the final separation of

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\* Arizona Copper Co., Ltd.

† Received Feb. 20, 1919.

the metal with completeness and commercial purity is an operation requiring skill and circumspection. How far this task has been solved at the New Cornelia is shown with commendatory minuteness in the paper under discussion. It is difficult to hold the composition of a commercial electrolyte within the limitations imposed by the requirements for high efficiency in the metal deposition and great care must be taken in its preparation.

It is gratifying to hear of the efficiency of sulfur dioxide as a reducer of ferric iron and of the negligible corrosion of hard-lead anodes, because predictions as to the impracticability of both have been frequently made. The regeneration of acid in the reduction process forms a valuable asset and, as regards the question of a serviceable insoluble anode for the electrolysis of sulfate liquors, we recognize that the subject has caused more agitation than was warranted.

It must be regretted that electrolysis will not practically lend itself to a more complete exhaustion of the copper in the discard liquors. We hear that it is doubtful economy to continue electro-deposition in solutions containing as much as 1.71 per cent. copper, by which time the ampere efficiency has dropped below 50 per cent.; and that precipitation by scrap iron is resorted to before this point is reached with the result that a good part of the output appears as cement copper.

The problem of profitable copper deposition, by electrolysis, of low-grade solutions is one of absorbing interest in the Southwest, where leaching plants will soon become a necessary addition to concentrators working up mixed ores. The radical difference between such a supplementary plant and the conditions existing at Ajo is the great dilution of the liquors, which cannot be separated from the leached pulp by draining and stage washing but only by dilution and decantation on account of the slimy nature of the product to be leached. A high concentration of copper in liquors from a material in which about 60 per cent. of the sizes are minus 200 mesh and frequently carry less than 0.5 per cent. copper is indeed impossible, if the separation of the liquor must be effected by settling and decantation; and the practice of leaching such a product by counter-current has as its object rather the economical use of the acid than a concentration of the liquor, except as far as an increased extraction raises the copper contents in the solution. In connection with this subject, it would be of interest to learn whether any progress has been made in the designing and application of real practical diaphragms. These have been proposed as a means for reducing the power consumption in the electrolysis of low-grade solutions. Any data bearing on this subject would be highly welcome. As far as I am aware, the difficulty is encountered in the creation of a durable diaphragm for solutions whose acidity increases with their depletion. If, without a diaphragm, the power consumption increases to 3 kw. hr. per pound of metal deposited from

solutions containing 0.5 per cent. of copper, there is but little hope that electrolysis will play a great role in the working up of liquors from leaching plants that are operated in continuity with the milling process. The treatment for recovery of the acid-soluble copper in weathered tailings does not come within the frame of my present consideration; they can be reclaimed in a comparatively dry state and treated with the advantages that result from this condition. Neither do I refer to any so highly oxidized material as would today constitute primarily a bad milling ore.

The high price of scrap iron in many localities, the expense of handling it, the high consumption in the precipitation from poor solutions, the large launder or tank equipment, and the production of a low-grade cement have each contributed in no small measure to bringing this method into disrepute. Therefore the positive statement of the authors predicting the early possibility of producing sponge iron successfully and at a price at which it can enter into favorable competition with scrap, will be welcomed by those who are in a position to take advantage of it and have themselves experienced the trials of an antiquated method.

J. W. RICHARDS,\* South Bethlehem, Pa.—It should be very comforting to those who are working with insoluble anodes that the anti-monial lead anodes, during a period of a year, had practically failed to show any marked deterioration, so that, so far as they are concerned, the very important problem of the unattackable anode seems to be solved. Another point worth attention is the use of the little conical, glazed, porcelain insulators to keep the anodes spaced properly away from the cathodes, a very simple device that apparently does its work satisfactorily.

FREDERICK LAIST,† Anaconda, Mont. (written discussion‡).—All who are interested in the treatment of oxidized copper ores by leaching have been awaiting the publication of this paper, which comes up to our expectations in all respects. Messrs. Tobelmann and Potter are to be congratulated upon the excellent way in which they have set forth the workings of the process as applied at Ajo.

Numerous attempts have been made to apply the electrolytic copper process but all have failed, except the installation of the Chile Copper Co., at Chuquicamata. Some of these failures were due to insufficient financial backing and inadequate ore reserves, which necessitated operations on too small a scale to be commercial, and to a lack of understanding of the scientific principles on which the success of the process depends. Those acquainted with the excellent organization that Dr. Ricketts has built up in Arizona felt that, once this process had been decided upon,

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‡ Received Mar. 10, 1919.

the successful outcome of the enterprise was a foregone conclusion; but few expected the excellent results obtained over so long a period.

The reduction of ferric solutions by means of sulfur dioxide has always been considered difficult, and experiments made by the Research Department at Anaconda tended to confirm this opinion. Another difficulty that seemed quite serious, and which has been most successfully overcome at Ajo, is the leaching of a low-grade ore with a solution comparatively high in copper, without, at the same time, leaving too much soluble copper entrained in the tailings. This is an important matter in view of the impossibility of electrolytically precipitating more than a comparatively small percentage of the contained copper during each cycle. The successful working out of a leaching process that enables good extractions to be made under so comparatively adverse conditions was no small part of the problem, and may well have taken more thought, study, and ingenuity than the electrolytic precipitation itself.

The writer had the pleasure, a few weeks ago, of visiting the plant and was greatly impressed by the way everything, both metallurgical and mechanical, had been foreseen and provided for. Excellent mechanical construction has had much to do with the smoothness with which everything operates. Every metallurgical engineer who has aided in the development of new processes knows that the best thought-out scheme may fail if the pumping system or the tank construction, or any one of the thousand necessary mechanical appliances, is defective. One is also struck by the forethought and consideration shown for the well-being of the plant operatives. The housing facilities are excellent and the civic center is a model of its kind and goes far toward making living conditions in the desert more pleasant than people accustomed to live in established communities generally realize.

H. A. TOBELMANN and J. A. POTTER (authors' reply to discussion\*).—Regarding Mr. Rose's discussion, we are of the opinion that the fouling of solution does not retard the dissolving power as much as is generally supposed. One of the principal objections is the difficulty in washing the tailings when leaching with a heavy solution. It has been shown that the lighter the solution, the more readily is the reduction with  $\text{SO}_2$ , the less the slime carried through the system, the purer the cathode copper produced, and the cleaner the tailings.

The suggestion regarding a higher copper content in tank-house electrolyte might prove to be a good one. This would necessitate many changes in our pipe lines and additional pumping capacity at the towers and could only be done after extensive experimental work along these lines.

In reducing the copper content from 4 per cent. to  $1\frac{1}{2}$  per cent. in the flow of the solution through the electrolytic tank house, an excessive quantity of ferric iron would be produced and poor tank-house efficiency would be the result. This trouble could be partly overcome by circulating tank-house electrolyte through the towers. This practice was followed for a time, but it was found that the absorption of  $\text{SO}_2$  gas was very low due to the high acid content of the electrolyte.

The advantages and the disadvantages of both motor generators and rotary convertors were extensively investigated and motor-generator sets were decided upon on account of their greater flexibility in obtaining the range in voltage considered necessary and maintaining at the same time a good average efficiency.

The suggestion to use a higher current density is good. We are now installing an additional motor-generator set, which will permit us to carry an average current density of 10 amp. per sq. ft. when necessary.

After six years of leaching experience, a certain leaching constant has been developed. It has been found that in ores of the same average copper content and for the same extractions the product of the total acid neutralized per ton of ore by the leaching period in days is a constant. By this we can govern the acid concentration.

It does not seem practical to us to increase the per cent. free acid in the solution going on the oldest ore, as we have definitely proved, other things being equal, that the higher the acid the greater is the quantity of impurities dissolved, consequently the more the discard of solution necessary and the greater the proportion of cement copper produced.

During February, this company, with the other copper companies, found it necessary to curtail production. This resulted in producing a number of unforeseen changes in operation, among which was that of a longer leaching period and operation with a solution of much lower gravity. Thus the leaching period time was increased, the acid concentration was correspondingly decreased, and the same average extraction was obtained.

The results were as follows:

	JAN.	MARCH
Tons ore leached per day, in tons.....	5513	3190
Copper in heads, per cent.....	1.374	1.414
Ore on 4 mesh, per cent.....	44.0	43.5
Copper in tailings excavated, per cent.....	0.268	0.276
Total copper dissolved, per cent.....	80.90	80.50
Average days of leaching.....	6.77	10.45
Average acid going on oldest ore.....	2.67	1.45
Average acid in solution coming off ore.....	0.38	0.36
Total pounds of 100 per cent. $\text{H}_2\text{SO}_4$ neutralized per ton of ore charged.....	67.4	45.0
Copper to launders, per cent. of total copper dissolved.....	24.5	11.1

Acid being one of the largest items of expense in this process, its economical use is essential. Aside from the question of cost there is the production of cement copper, which under present conditions costs considerably more per pound than electrolytic. Thus in our process the greatest profit can probably be obtained by the use of the minimum acid concentration for a maximum time.

Elko Prince Mine and Mill

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MIDAS, NEV.

(Colorado Meeting, September, 1918)

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GEOLOGICAL CONDITIONS

THE Elko Prince mine is in the Gold Circle district, Nevada, about 1½ miles (2.4 km.) from the town of Midas, 55 miles (88.5 km.) west of Battle Mountain and 50 miles (8.5 km.) northeast of Golconda. The district is described by Emmons in U. S. Geological Survey *Bulletin* 408, 1910. Little was said of the Elko Prince, however, as only a small amount of work had then been completed.

The geological structure of the region is simple. The oldest exposed formation, covering most of the area, is rhyolite, which has a light-colored devitrified groundmass enclosing phenocrysts of feldspar, quartz, and magnetite. It has been somewhat weathered and partially stained with oxide of iron. There are several outcrops of andesite which cut the rhyolite. The ore deposits occur as fissure veins, replacement veins, and sheeted zones. The Elko Prince vein is of the first type. The rhyolite near the veins is silicified, stained with oxide of iron, and, at some localities, replaced by ore, or is so permeated by metals as to be classed as ore. The vein filling consists chiefly of quartz, and the values consist entirely of gold and silver. Part of the gold exists as the native metal and part is associated with pyrite. Silver occurs free and as argentite in banded streaks through the vein. These veins trend in a northwesterly direction, with dips varying from 65° to vertical.

DEVELOPMENT OF ELKO PRINCE PROPERTY

Gold was discovered in the district in the Fall of 1907, and a characteristic rush followed. The town of Midas was laid out and shortly had

\* President, The Dorr Co.  
† General Superintendent, Elko Prince Mine.

2000 inhabitants. Several small mills were built, one of which, the Rex, ran several years. After the first excitement, the people drifted away rapidly, and at present only about 150 remain. The Elko Prince claims were located in 1907 by Paul Ehlers, a prospector, and 10 claims were patented in 1913 after considerable development work had been done. The property was purchased in 1908 by the Elko Prince Mining Co., L. L. Savage, President, and was extensively developed between 1911 and 1915.

The outcrop of the Prince vein is insignificant, only a narrow seam of low-grade quartz, or an obscure fissure containing no quartz or metals. The first work on the property was a 60-ft. (18-m.) shaft sunk on the cropping. A narrow seam of mineralized quartz was followed, but nothing of importance was found; then a 240-ft. (73-m.) drift on the vein gave similar results. Later, a 700-ft. (213-m.) crosscut reached the vein 270 ft. (82 m.) below surface, and cut an ore shoot almost in the center. Subsequent work developed a vein having an average width of 14 to 15 in. (35.5 to 38 cm.), a total tonnage of 30,000, and a gross value of approximately \$1,000,000, the limits of the orebody being quite well defined.

In June, 1915, the Elko Prince Mining Co. made an agreement with the Dorr Co., whereby the latter was to design, finance and build a cyanide mill of at least 40 tons daily capacity, furnish additional mine equipment, and operate the property until certain financial results had been attained. Milling was begun in November, 1915, and the original terms were completed in June, 1917, but the Dorr Co., operating through the Elko Prince Leasing Co., has continued in charge of the property to the present time.

#### PRINCE VEIN

The Prince vein has a uniform northwest-southeast strike and a dip varying from vertical to 85° to the northeast. One horizontal fault of a few feet occurs in the 1400 ft. (426 m.) of development on the property, and a second, of much greater extent, probably occurs just beyond the south end line.

The vein filling is hard but brittle quartz, with a very small amount of sulfides. Partial oxidation extends from the surface to the deepest openings. Manganese in oxidized form is found in spots. No enrichments due to leaching and redeposition have been observed, except as to a small portion of the silver. The gold is mainly free but very fine; about 50 per cent. can be amalgamated. Thin flakes of native silver are found in the footwall and penetrate its seams in places to a depth of an inch. The silver exists chiefly as argentite and polybasite, giving the quartz the characteristic banded appearance that usually accompanies the presence of these minerals.



A characteristic analysis of the ore is:

	PER CENT.		PER CENT.		PER CENT.
SiO <sub>2</sub> .....	85.0	S.....	0.1	Ignition loss.....	2.0
Al <sub>2</sub> O <sub>3</sub> .....	2.0	As.....	0.1	Cu.....	0.0
Fe.....	3.0	Sb.....	0.1	Au (oz. per ton)...	0.7
CaO.....	4.0	Mn.....	Trace	Ag (oz. per ton)...	10.0

A second vein, the June Bell, is parallel to the Prince, and lies a short distance to the west. It is of the same type but much smaller, and gives promise of producing but little metal.

The development of the Prince vein at the time milling began is shown in Fig. 1, the dotted lines indicating the extent of ore estimated

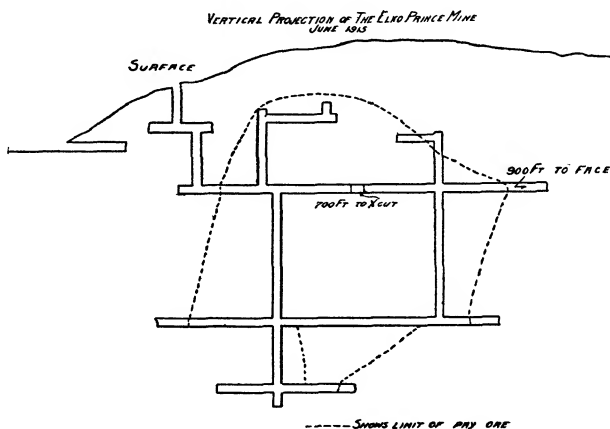


FIG. 1.—DEVELOPMENT OF THE ELKO PRINCE VEIN, JUNE, 1915.

as profitable. Above the 300-ft. (91-m.) or main level, mining has confirmed the accuracy of the estimate. Below the 300-ft. level, the orebody has been proved to extend slightly further to the north, considerably further to the south, and some little ore has been found below the 600-ft. level.

## MINING PRACTICE

### *Stoping*

The following conditions influence the method of mining. The vein is almost vertical; its average width is 15 in. (38 cm.), seldom less than 12 or more than 24 in. thick; the west wall is smooth, hard, and firm, with quartz sometimes frozen to it; the east wall is less well defined, decomposition having extended into it to a depth of a few inches to two or three feet; a sliding movement has occurred in the east side of the vein, and at some points a considerable amount of gouge is found, containing pieces of vein matter.

When planning a method of mining, stripping (resuing) was considered, the idea being to mine the east wall as narrow as possible, use it for filling, break the ore away from the west wall, and run it all out. The vein, however, was too brittle in many places to permit this; all of the quartz in the gouge would have been lost, as well as much of the vein proper. The advantages of this method—the small amount of high-grade ore to be milled, and the smaller plant to be erected—were more



FIG. 2.—A STOPE BETWEEN THE 450 AND 600-FT. LEVEL.

than offset by the greater cost of mining and the inevitable losses. Stopping the ore with some waste, as little as possible, required a plant of 25 to 50 per cent. greater capacity, and involved a small increase in power and supplies consumed, but this method permitted the recovery of all of the ore, and it was thought that the increased milling expenses would be less than the loss by any selective method of mining.

Overhead stoping was adopted, enough ore being drawn daily to maintain proper working room above the broken ore. No sorting was attempted in the stopes. In spite of the fact that the stopes were almost

vertical, and that one wall was hard and smooth, the ore would occasionally hang and cause trouble. After a time, it was found that if a little ore was drawn daily from every shoot, and the broken ore was not permitted to remain at rest for a whole day, it would remain loose enough to run freely.

Predictions as to the most efficient stoping width ranged from 30 in. to 48 in. (76 to 121 cm.); it has been found possible and practicable to maintain an average stope width of 32 in. throughout the mine. Above the 300-ft. level, the average was 28 in.; in many places, the miners broke only 20 in., but the ore would not draw freely through such narrow stopes. Fig. 2 shows a typical stope, with good walls.

At the north end of the shoot, where the ground was quite dry, the stope was carried from the 600 right through to the 300 level. At the south end, where rather wet, an intermediate level at 450 was found advisable.

### *Drilling*

Five 16 V Waughs, supplied with 1 $\frac{1}{8}$ -in. (28.6-mm.) cruciform steel, are used in stoping. Drifting and sinking are done with Waugh clipper drills using  $\frac{7}{8}$ -in. (22.2-mm.) hollow steel. A pressure of 85 lb. of air is maintained at the drills.

### *Contract Stoping*

Mining was first done on straight day's pay; but, within a few months, a bonus system was adopted whereby a premium, increasing with each 5 ft. (1.5 m.), was paid on all footage drilled in excess of 50 ft. per day. Before initiating that system a careful record was kept of the work of each man for two months, in order to secure a basis for a just bonus. It was understood that no change in unit prices would be made thereafter, regardless of the amount that might be earned. It is most important for the success of any bonus system that the men shall recognize that the whole saving, as compared with day's labor, is to be divided with them, and that the system is not to be designed for the purpose of reducing them to the standard wages of the camp. It was found that some men, on contract, would break twice as much ground as when on day's pay. There was some tendency to place holes too close together if the ground was easy for drilling. Straight contracting was finally adopted, based on the square foot of vein area broken; the contractors are obliged to keep the walls free from ore and maintain the stopes within limits designated by the foremen. The company furnishes the powder and the men do the shooting. The usual price in such contracts is from 12 to 15 c. per square foot, at which rate the men make from 25 to 75 per cent. more than day's pay.

### *Loading and Tramming*

The chutes on each level are spaced about 27 ft. (8 m.) apart, and are equipped with the gate shown in Fig. 3. By having the gate boards extend across the end of the chute, they can be knocked open at either side, without binding.

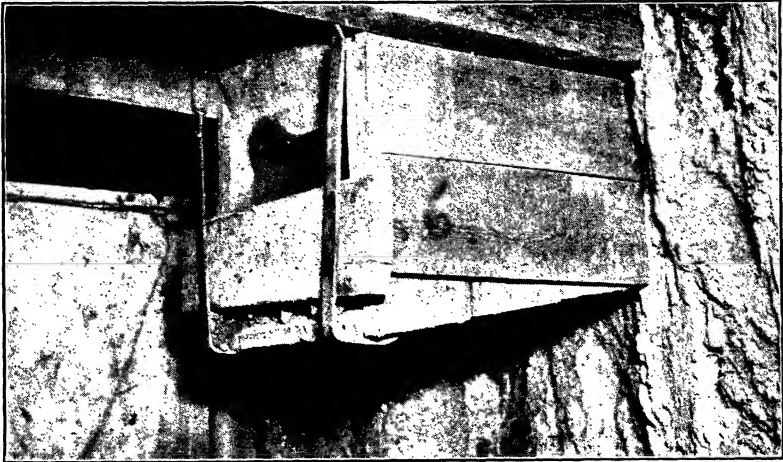


FIG. 3.—AN ORE CHUTE.

The mine cars are 30-cu. ft. (0.85 cu. m.), Truax side-dump cars weighing 1100 lb. One man and a horse readily handle 60 tons a day with an average haul of 1100 ft. (335.28 m.), using a five-car train from the 300-ft. drift to the mill bins. Hand tramming is done on the lower levels, two men handling the full tonnage.

### *Hoisting*

The selection of hoisting equipment was determined by the following considerations:

1. The shaft followed the changing dip of the vein, making rapid hoisting dangerous.
2. It was expected that, with the size of engine available for the power plant, this would be fully loaded, making a high load factor imperative.
3. With the small tonnage, the saving of a single man meant a large cut in the cost per ton.

A Denver Engineering Works' double-drum electric hoist, with counterweight, was selected. It is driven by a 10-hp., 550-volt, d.-c. motor, and has a hoisting speed of 150 ft. per minute. The complete cycle takes 5 min. from the 600-ft. to the 300-ft. level. The self-dumping skip weighs 1200 lb., and has a capacity of 35 cu. ft. The trammers dump their cars directly into the skip through a chute.

*Timbering*

When the mill started, the need of ore required drawing faster than shrinkage stoping allowed, and the miners were obliged to work on stulls. Trench jacks were found advantageous for this purpose, as they could be removed before shooting and put back into place much quicker than stulls. The main timbering now consists of chutes, manways, and stulls in the stopes as the ore is drawn. The wet, sticky nature of the ore in the narrow stopes causes the ore to pull nearly vertical over the chutes, and thus requires close stulling, but light pieces, such as 3 by 4's or 4 by 6's, are sufficient in most places. Very little timbering is required in the drifts.

*Cost of Mining*

Mining costs for 1917 are given in Table 1, covering a total of 21,674 tons, or 59.4 tons per day. The costs do not include prospecting for additional orebodies.

TABLE 1.—*Mining Costs per Ton for 1917*

Timbering and chutes.....	\$0.5588
Blacksmith.....	0 0995
Stoping.....	0.8083
Tramming.....	0.2822
Hoisting.....	0.1956
Air.....	0.1288
Powder, caps, and fuses.....	0.4452
Repairs.....	0 0486
Drill steel.....	0 0570
Miscellaneous .....	0.4987
Total.....	\$3 1227

Note: Miscellaneous includes local supervision, assaying, insurance, taxes, and sundry miscellaneous items.

## MILLING PRACTICE

*General Design*

The financial arrangements allowed the mill to be designed so that when the orebody already developed had been worked out and the plant scrapped, the greatest net profit would have been made. The treatment of 50 tons of sorted ore daily promised a life of about  $3\frac{1}{2}$  years, with the chance of an extension by finding other orebodies or obtaining custom ore.

Labor was scarce in the district, and, although there was no labor union, the Tonopah scale of \$4 minimum for 8 hr. had been paid.

Redhouse, on the Western Pacific, the nearest freight station, is 34

miles away, and hauling by wagon costs from \$13.50 to \$15 per ton. The road from Redhouse to Midas is a good desert road, with a large increase in grade in the last 5 miles. The chance of heavy snow at the upper end in Winter, and the certainty of very heavy hauling in Spring, make it essential to stock up by December to run until May. Hauling by team has proved more satisfactory than by motor truck. Oil storage of 22,000 gal. was provided at the mill. No electric power was available.

Although the ore is a hard quartz, a variable quantity of very slow-settling gouge occurs with it, so that it was necessary to provide a large

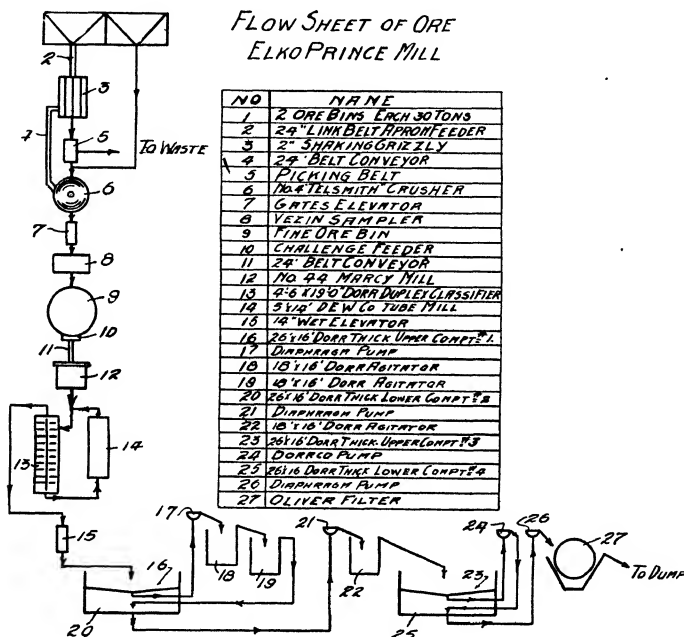


FIG. 4.

settling area per ton. The treatment, shown in Figs. 4 and 5, follows the standardized lines of ball-mill crushing and fine grinding, with counter-current decantation and zinc-dust precipitation. As this pulp cannot be thickened to less than 60 to 65 per cent. moisture, a dewatering filter is used to enable the tailings to be conveniently handled, and to reduce mechanical losses of cyanide and metal.

Fig. 6 shows a plan of the mill, with many of the details omitted, but with flooring and runway elevations indicated; Fig. 7 shows one elevation. It will be noted that the working floors are nearly at one level, and that the machines requiring close attention, such as the Diesel engine, the filter, etc., are grouped near the center of the main building, where they

can be under observation from any point where the operator must work. This arrangement, feasible only in a small plant, enables one man to keep general watch over the whole operation of fine grinding and cyaniding, as well as the power plant for mine and mill; so that, while two men are used on the two night shifts as a matter of precaution, the mill man frequently sends his helper to the crusher room for a few hours, when short of ore. When not so required, the shift men do a good deal of repairing, and prepare the samples for the assayer.

The relative elevations of the fine-grinding and cyaniding departments are of interest. If the objection frequently urged to the elevation of pulp had been considered, and a gravity flow from the classifier to the first thickener had been attempted, the cost of the excavations and the building would have been much greater, the convenience of operating dimin-

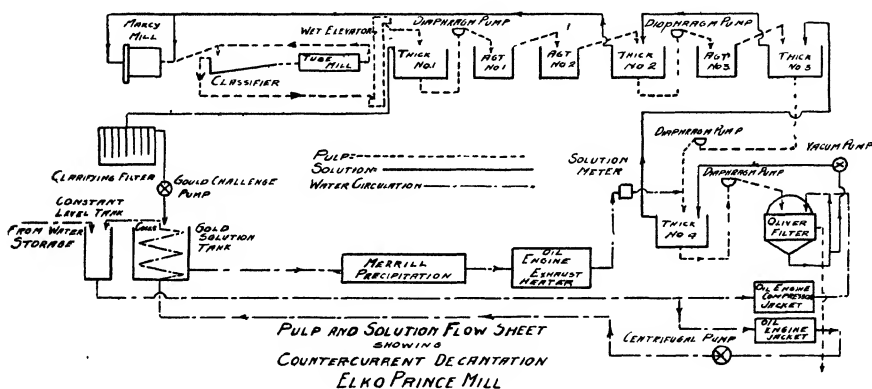


FIG. 5.

ished, and additional pumping of clear solution would have been needed. We consider a well designed wet elevator to be at least as efficient as a pump.

Except this one elevation of combined ore and solution, it will be noted that in all elevations some other necessary purpose is accomplished as well. For instance, the diaphragm pumps, transferring the thick pulp, also serve to regulate its discharge; and the clarifying-filter and precipitation-press pumps serve to elevate the solution to the last and highest thickener, from which it flows through the others and through the grinding system back to the elevator.

The use of the closed-type Dorr tray thickener (Fig. 9) with the flow in series, makes each tank do the work of two, and cuts the necessary settling-floor space in half. The advantage of saving mill space in this case is unusually important as coal costs \$26 per ton and the waste heat from the oil engine is entirely relied on to heat the mill.

Normal operation requires only one storage tank (the gold tank) for solutions, as No. 3 thickener serves as a mill-solution supply tank, and the barren solution flows directly to No. 4 thickener. There should be, however, a sump or reserve tank large enough to take the contents of a thickener, to avoid possible loss of solution in case of an accident; such a tank could be outside the mill.

### *Coarse Crushing*

Two 10 by 12-ft. (3 by 3.6 m.) receiving bins, with 50° bottoms lined with No. 10 steel, hold 30 tons of ore each. No. 1, located only 60 ft. (18 m.) from the mouth of the tunnel, is covered with a grizzly made of 30-lb. rail, with 7-in. (177.8 mm.) spacing. It has a 24-in. (60.9 cm.) Link-belt feeder discharging to a shaking grizzly. The undersize goes to the elevator by belt conveyor; the oversize goes to an 8 ft. by 30-in. Jeffrey steel-pan picking belt, traveling 3 ft. per min., then to a No. 4 Tel-smith gyratory crusher. From 5 to 10 tons of \$2 waste per day is sorted out. No. 2 bin, used for custom ore, discharges directly into the crusher. The crusher has given good service; the chilled-iron mantle lasted two years, and the concaves are still good. We believe that the installation of this more expensive crusher equipment, which is standard for large mills, is well worth while even at a small mine, on account of the saving of labor and the uniform crusher feed; one man usually crushes and sorts the whole tonnage. The Gates dry elevator, at 55° slope and belt speed of 120 ft. (36.5 m.) per min., has been very satisfactory on the wet, sticky ore of the mine.

A Vezin sampler, with intermediate crushing to  $\frac{1}{4}$  in. (6.35 mm.) through a Samson crusher, after the first cut, is used on custom ore and to check feeder samples on company ore. It cuts out  $\frac{1}{5}$ ,  $\frac{1}{10}$ , and  $\frac{1}{10}$ .

The crushed-ore bin is a 15 by 15-ft. (4.57 by 4.57 m.) steel cylinder on a wood platform, having an 18-in. (45.72 cm.) center discharge to a Challenge feeder, which delivers to a 12-in. belt conveyor leading to the Marcy mill. An opening in the side of the bin, at the bottom, about 3 ft. high and 30-in. wide, and extending like a tunnel about 4 ft. toward the center so that no ore will flow out, allows access to the inside of the bin for loosening sticky ore.

### *Fine Crushing*

The Marcy mill discharges to a Dorr classifier in closed circuit with a tube-mill, the classifier sands being returned to the tube-mill by a screw conveyor. The solution overflowing No. 2 thickener is divided between the Marcy mill, the classifier, and the tube-mill. For satisfactory extraction a product containing not more than 1 per cent. coarser than 100 mesh, and at least 78 per cent. finer than 200 mesh is required.



TABLE 2.—*Fine-crushing Data*

MARCY MILL	TUBE-MILL
Size, No. 44	Maker, Denver Eng. Works
Speed, 36 r.p.m.	Size, 5 by 14 ft.
Ball load, 2200 lb.	Speed, 23 r.p.m.
Size of balls, 4 to 5 in.	Pebble load, 14,000 lb.
Maximum size of feed, 2 in.	Size of pebbles (Danish flint), 4 in.
Maximum size of discharge, $\frac{1}{4}$ in.	Rate of crushing, 133 tons per day
Rate of crushing, 54 tons per day	Power, estimated, 35 hp.
Power, estimated, 25 to 35 hp.	Water in feed, 35.1 %
Water in feed, 39.4 %	Scoop feeder, 30 in. radius
Liners, manganese steel	Liners, Komata
Grates, chrome steel	

DORR CLASSIFIER

Model "C" Duplex  
Size, 18 ft. 8 in. long by 4 ft. 6 in. wide  
Slope, 2 in. per foot  
Speed of rakes, 14 strokes per min.  
Depth of tank at overflow, 21 in.  
Water in discharged sand, 34.3 %  
Water in overflow, 8.6 parts to 1 part solids

SCREEN TESTS

Mesh	Marcy Mill Discharge		Tube-mill Discharge		Classifier Feed		Classifier Discharge		Classifier Overflow	
	%	Cum. %	%	Cum. %	%	Cum. %	%	Cum. %	%	Cum. %
Over 10..	15.5	15.5	.....	.....	7.4	7.4	9.1	9.1		
10- 20	17.0	32.5	0.2	0.2	9.4	16.8	7.9	17.0		
20- 65	28.1	60.6	9.1	9.3	17.7	34.5	26.8	43.8		
65-100	5.9	66.5	15.6	24.9	12.0	46.5	19.4	63.2	1.0	1.0
100-150	7.0	73.5	26.6	51.5	16.2	62.7	23.5	86.7	4.1	5.1
150-200	4.4	77.9	13.6	65.1	8.9	71.6	8.5	95.2	13.4	18.5
Below 200	22.1	100.0	34.9	100.0	28.4	100.0	4.8	100.0	81.5	100.0
	100.0		100.0		100.0		100.0		100.0	

COSTS, CENTS PER TON

MARCY MILL

Balls (1.13 lb.) .....	9.59
Liners.....	6.03
Repairs.....	4.16

CLASSIFIER

Repairs.....	0.03
--------------	------

TUBE-MILL

Pebbles (3.50 lb.) .....	6.84
Liners.....	1.52

The Marcy mill (one of the earliest types) as first installed had an open scoop feed and iron-lined wooden scoop box, but this has been replaced by a self-contained scoop with end feed, which gives much better results. Cast-iron feed and center liners were unsatisfactory, and the mill is at present equipped with manganese-steel liners and a chrome-steel grate.

The tube-mill is equipped with Komata shell liners composed of manganese-steel angle bars and cast-iron filler bars and plates; the end liners are cast iron. The operation of the mill has been entirely satisfactory in every way, and the cut gears give excellent service. The life of the liners is about 18 months; the end liners last about one year.

No repairs have been required for the classifier, except the renewal of the screw conveyor flights, which last six months.

### *Wet Elevator*

This is a standard elevator composed of 14-in. (35.56 cm.) 7-ply rubber belt, and runs at 380 ft. (115.8 m.) per min.; 12 by 6-in. Salem buckets, of No. 14 steel, are spaced 12 in. center to center. The boot is concrete, and large enough to retain the contents of the buckets in case of a spill. The lower journals are one-half bearings, mounted in the boot but outside the housing; they are maintained at constant tension by a counterweight, and are hinged on a shaft at the back of the elevator. The oil-soaked hardwood blocks last 8 months. This boot is a design developed by N. Cunningham at the Hollinger mill, and is most satisfactory. Slipping of the belt developed as it wore, and was eliminated entirely by attaching six steel strips  $\frac{1}{2}$  by  $\frac{3}{32}$  in. (12.7 by 2.38 mm.) across the face of the head pulley; the strips do not injure the belt, and probably will increase its life by preventing slippage. The first belt lasted two years, and the life of the buckets working on this fine pulp is indefinite, as the tendency to coat with lime balances the wear.

### *Tray Thickeners*

The closed-type Dorr tray thickener is shown diagrammatically in Fig. 9. Wooden tanks 26 ft. (7.9 m.) diameter by 16 ft. (4.8 m.) stave are used and the trays are of wood, following the earlier practice. In this type the tray divides the tank into two separate compartments, with no circulation or leakage of pulp between them, the central shaft being suitably packed where it passes through the tray. Both compartments are fed, discharged, and overflowed independently, and thus two distinct operations are carried on at once. For instance, in the top compartment of No. 1 primary thickening takes place; while in the bottom, a thickening of the agitated pulp after a second dilution is accomplished.



### Diaphragm Pumps

Two duplex, 4-in. center-valve pumps take the discharge from the thickeners. They have an adjustable stroke and make 50 strokes per minute. Very close adjustment is made by admitting air to the suction. The diaphragms last from 4 to 6 months.

### Agitation

Three standard Dorr agitators are used in wood tanks 18-ft. diameter by 16 ft. high. Each gives 24 hours' agitation with normal tonnage and dilution. They operate at 3 r.p.m., and only enough air circulation to prevent segregation is needed. Drag chains on the distributing

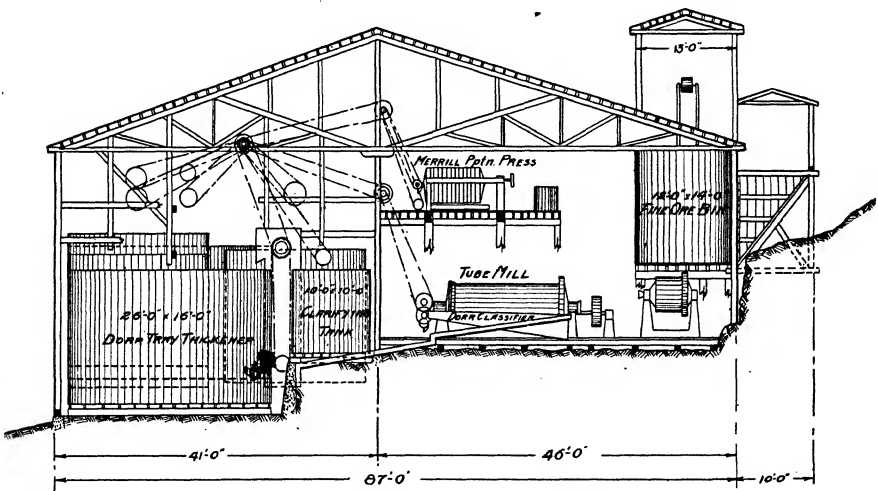


FIG. 7.—A SECTION THROUGH THE MILL.

launders prevent building up of solids on the tank sides. A 9 by 6-in., 100-cu. ft., low-pressure, Ingersoll-Rand, Rogler type compressor supplies air for the three agitators, the solution meter, and for discharging the Oliver filter.

### Filtration

An Oliver filter,  $11\frac{1}{2}$ -ft. diameter, 10-ft. face, is used for final washing and dewatering. It makes a revolution in 8 min., and a 14-in. vacuum, maintained by a dry vacuum pump, gives a  $\frac{1}{16}$ -in. (11.11-mm.) cake. The pulp is fed to the filter near the bottom of the tank, maintaining a zone of clear barren solution above the pulp, which assists in washing. Barren solution wash is used on the rising side, and water on the descending. The cost for repairs has averaged 3.52 c. per ton.

*Solution of Metals*

The mill-feed assays from 0.6 to 1 oz. Au, and from 7 to 14 oz. Ag per ton. Figures for March, 1918, taken as typical, show that, on the basis of bullion plus tailings, there was an extraction of 97.5 per cent. of the gold and 87.1 per cent. of the silver. These figures are representative of results obtained on Elko Prince ore; but, in the treatment of custom ore, variations in extraction and chemical consumption are large. Table 3 shows the distribution of the extraction among the different units in the process. The figures were obtained by sampling over a period long enough to eliminate individual errors.

TABLE 3.—*Extraction, Per Cent.*

UNIT	GOLD	SILVER
Tube-mill.....	80.0	48.3
Thickener No. 1.....	3.5	8.2
Agitator No. 1.....	9.0	22.2
Agitator No. 2.....	2.0	3.1
Thickener No. 2.....	0.7	0.8
Agitator No. 3.....	0.8	3.0
Thickener No. 3.....	0.6	0.2
Thickener No. 4.....	0.3	0.5
Oliver Filter.....	0.6	0.8
Total.....	97.5	87.1

The benefit from change of solution is more evident on the silver than on the gold; the silver also shows the effect of contact with a solution diluted by the fresh water added at the filter. It has been a common experience, both in sand leaching and filtration, that when the commercial extraction has been obtained, and a final displacement water wash is added, a further amount of metal dissolves just when it cannot be economically recovered. The use of excessive wash water, and its precipitation before wasting, reduce this loss.

Silver at \$1, instead of 50 c. per ounce, as when the mill was built, requires a careful study of the chance for increasing extraction, but capital expenditures now are necessarily curtailed by the high cost of equipment and by the limited amount of ore remaining in the mine. An additional tray thickener would give a slight increase in solution and a decrease in dissolved loss. The solutions now are heated only by waste heat, but it is possible that direct heat, in spite of its cost, would be profitable. Lead acetate was used first, but was replaced by litharge with equal results and lower cost; about 1.5 lb. per ton of ore gives the most economical results. Working tests have not indicated that finer crushing or longer agitation would be profitable.

The dissolved loss depends on the grade of the ore and the efficiency of precipitation. While the barren solution can be maintained at 4 c. or

less, the loss is below 8 c. per ton (Ag at \$1); but, if precipitation gives trouble and the barren solution goes to 4 to 8 c., the loss jumps to 8 to 15 c.

Cyanide is added to the tube-mill feed, the strength being maintained at 2.3 lb. NaCy per ton of solution; the strength in the agitators is 2 lb. NaCy. The mechanical loss of cyanide is about 0.6 lb., and the chemical consumption about 0.5 lb. per ton of ore. Lime is maintained at about 0.6 lb. per ton of solution in the tube-mill and about 0.4 lb. per ton of solution in the agitators.

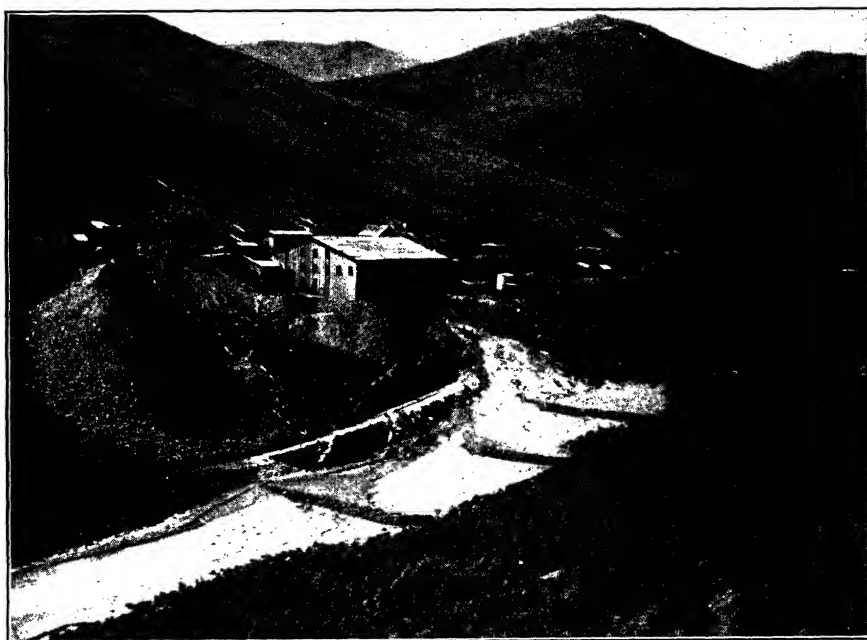


FIG. 8.—VIEW OF THE ELKO PRINCE PROPERTY.

The consumption of materials is tabulated in Table 4. It has recently been found possible to reduce the lime, and profitable to increase the litharge.

TABLE 4.—*Consumption of Reagents, Pebbles, and Balls in 1917*

	LB. PER TON OF ORE	COST PER TON OF ORE
Sodium cyanide.....	1.10	\$0.2866
Lime.....	11.71	0.2152
Pebbles.....	3.50	0.0684
Balls.....	1.13	0.0959
Zinc.....	1.08	0.2093
Litharge.....	0.44	0.0598

*Clarifying*

The overflow from thickener No. 1 goes to the clarifying filter, which is in a 3-in. (76-mm.) redwood tank, 10-ft. (3-m.) diameter by 10 ft. high. Across the top of this tank a 3-in. header carries connections for 24 filter leaves, only 18 of which are in use. Each is  $5\frac{1}{2}$  by 4 ft. and made with 12-oz. canvas. The leaves are connected to the header by short lengths of  $\frac{1}{2}$ -in. iron pipe,  $\frac{3}{4}$ -in. hose and Butters quick opening clamps. A Gould 7 by 6-in. Challenge pump draws the solution through the filter and discharges it into the gold tank.

*Precipitation*

The amount of solution precipitated daily is 280 tons as measured with a Tanner meter; pregnant solution assays from \$3 to \$5 per ton, and the barren solution from 2 to 6 c. A Merrill precipitation press with 14 frames, 52 by 3 in., is used with a Merrill zinc-dust feeder, and operated by a Deming triplex pump.

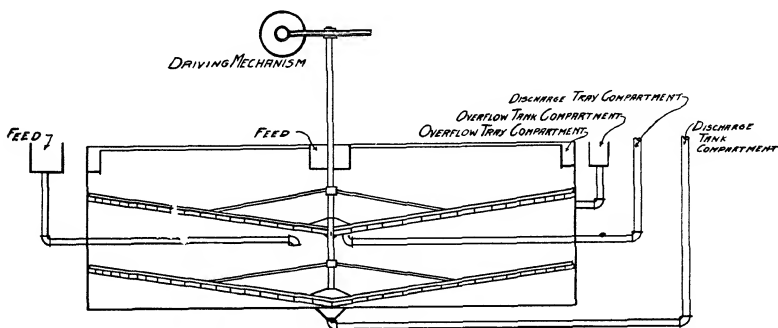


FIG. 9.—DIAGRAMMATIC DRAWING OF DORR CLOSED-TYPE TRAY THICKENER. (Patented.)

Both Merrillite and Colorado zinc dust have been used, and it appears that, under the Elko Prince conditions and costs, the latter is more economical. The cost for zinc dust during 1917 was \$0.2093 per ton of ore milled, or approximately \$0.04 per ton of solution precipitated; the actual consumption of zinc dust was about 0.212 lb. per ton of solution precipitated, or 0.12 lb. per ounce of bullion produced. The Crowe vacuum precipitation equipment, controlled by the Merrill Metallurgical Co., has recently been installed and the results of the first 2 months show a saving in zinc dust of about 50 per cent.

*Refining*

The press is cleaned up once or twice a month. Acid treatment is unnecessary, as the dry precipitate contains 75 to 85 per cent. gold and silver and gives a bar 950 fine. Drying to 15 per cent. moisture is done in an oven heated by the exhaust gases of the Diesel engine. A press,

made by the Illinois Supply & Construction Co., forms the product into briquets of 3 in. diameter and weighing 2 lb. each, at the rate of 12 per minute.

Melting is done in a Duplex Monarch tilting furnace with Lindsey burners. The flux contains 12 of nitre, 8 of borax, and 4 of silica, and the usual clean-up handles 2000 lb. of briquets, yielding 1500 lb. of bullion.

### *Heating*

The mill is heated entirely by radiation from the engine and solutions, and can be maintained at a comfortable temperature through zero weather. The barren solution is heated by passing through a Williams Tool Co. heater, connected to the engine exhaust, on its way to thickener No. 4. Additional heat is obtained by circulating the engine and compressor jacket water through the gold tank, and by using water at 90° F. from the Diesel engine and compressor for replacement water in the filter. This maintains a temperature of 75 to 85° F. during treatment, at no cost.

### *Water Supply*

Water is pumped from a well below the town of Midas, a distance of 11,000 ft. with a lift of 700 ft. A crude-oil engine was first installed, but it required so much attention that it was replaced with a 10-hp. motor; the water pipe is used as the return electrical circuit.

### *Cost of Milling*

Some of the milling costs for 1917 have been given before. A complete statement is shown in Table 5.

TABLE 5.—*Cost per Ton for Milling 20,087 Tons in 1917*

	Repairs		Operating		Totals
	Material	Labor	Supplies	Labor	
Crushing and sorting.....	0.011	0.029	0.010	0.142	\$0.192
Grinding and cyaniding....	0.177	0.112	0.172	0.305	0.766
Chemicals.....			0.771		0.771
Power (a).....	0.040	0.038	0.355	0.090	0.523
Water.....			0.037	0.015	0.052
Assaying and sampling.....			0.027	0.093	0.120
Office and supt.....			0.040	0.150	0.190
Insurance and taxes.....					0.113
Refining.....			0.045	0.025	0.070
Miscellaneous operating.....			0.020	0.035	0.055
Miscellaneous repairs.....	0.031	0.139			0.170
Totals.....	0.259	0.318	1.477	0.855	\$3.022

(a) The charge for power includes fuel and lubricating oils, one-half the time of millman, and all repair labor and materials. It is allocated to milling, water, mine air, hoisting, and mine development, according to estimated power distribution.



## POWER PLANT

As electric power was not available, and coal was too expensive, an oil engine was the only motive power considered. A 150-hp. Fairbanks-Morse engine on the property might have furnished a large part of the power needed; but, as fuel oil cost 12 c. per gal. delivered, and the rated consumption of the engine was double that of a Diesel, the initial saving would have been lost in operating.

The choice finally lay between an Allis-Chalmers Diesel and a De la Vergne semi-Diesel; the Diesel was selected. While both are excellent engines, one point of comparison between the two may interest possible users. The Diesel feeds the charge of oil, against no pressure, into a cup from which it is atomized by 700 lb. air, while the semi-Diesel sprays a mixture of oil and air, against heavy pressure, into the cylinder. It has proved possible to keep a valve tight, discharging air alone under high pressure, for 6 to 12 months; whereas, when spraying a mixture of air and oil, the makers only claim that the valve should remain tight a month, and our experience at another plant showed that, on account of loss of efficiency when they begin to leak, it is advisable to regrind valves every 7 to 10 days.

First cost, economy of attention, and saving of waste heat suggested one unit for mine and mill, with a small gasoline engine for circulation of the pulp in case of a shutdown. The mill plan shows the main line shaft driving all the heavy equipment, including the tube-mill, Marcy mill, mine compressor, crusher, hoisting dynamo, and the dry vacuum pump for the Oliver filter. A parallel countershaft drives, through a bevelled gear, the slow-speed line shaft for the thickeners, classifier, agitators, diaphragm pumps, elevator, etc.

The engine has a rating of 180 hp. at sea level, or 145 hp. at the altitude of the mill. It has two 12 by 24-in. cylinders, and requires about 170 gal. of 24° Bé. oil per day. Ample lubrication has been found essential. The oil filter furnished with the engine was replaced by a De la Vergne oil reclaimer which has given much better satisfaction, entirely eliminating the free carbon in the oil, so that twice the amount is fed to the bearings, with less consumption.

It was found that the best temperature for the circulating water in the engine cylinders was 110° F. This proved entirely too high for the best results in the compressor, as the lubricating oil carbonized rapidly, so that a separate circuit, discharging water at 90° F., was installed for the compressor.

It has not been possible to measure the power output per gallon of fuel used, but as the engine radiation, the jacket water, and the exhaust are all used to heat the mill and solutions, the total efficiency in winter must be very high. The exhaust gases have been frequently as low as 110° F.

## CONSTRUCTION COSTS

The following figures, representing costs of construction in 1915, are, unfortunately, only of historical value to-day.

Mill, complete (including power-plant).....	\$67,509.06
Refinery .....	2,560.11
Oil storage and wagons .....	2,162.05
Office, assay office, and other buildings and equipment .....	8,071.48

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Total surface expenditures, except water line already installed. \$80,302.70

The total cost of the mill machinery and tanks, at shipping point, was \$33,647.18, checking rather closely the common estimate that a mill erected will cost double the cost of its machinery.

The lumber, 205 M. costing \$48 per M. at the site, cost \$20.42 per M. to erect. Machinery, 145 tons, cost \$22.40 per ton to install; and 10 tons of piping was placed for \$44.77 per ton.

## Fine-grinding Cyanide Plant of Barnes-King Development Co.

BY J. H. MCCORMICK,\* MARYSVILLE MONT.

(Colorado Meeting, September, 1918)

THIS plant, near Marysville, Mont., was planned to treat the ore from the Piegan and Gloster mines, the latter being one of the early and famous producers of the Marysville district. When the mill was closed in 1888, treatment consisted of stamp milling, followed by pans and settlers for pan amalgamation. The extraction was evidently poor, because, a few years later, thousands of tons of tailings were re-treated by the ranchers in the valley below the mill, by the then new cyanide process, and gave a handsome profit.

In 1914, the present owners, after laboratory tests and an experimental mill run, decided upon the following treatment: crushing to 40 mesh in cyanide solution, concentration, amalgamation, classification, and cyanidation of sands by leaching, and of slime by agitation and decantation in charges. The ore averaged about \$7 per ton, gold and silver, the ratio of weight being 1 oz. gold to 7.56 oz. silver. The ratio of gold and silver in tailings was 1 oz. Au to 36.6 oz. Ag; the quartz was sharp and sandy, however finely ground, and rather difficult to slime.

The mechanical equipment was a No. 5 Symons gyratory crusher; three 10-ft. Lane slow-speed Chilean mills; one Wilfley roughing and one finishing table; one submerged-type Akins classifier; five 26 by 10-ft. fir leaching tanks; one 24 by 7-ft. Dorr thickener; four 14 by 16-ft. Dorr agitators; four 6-compartment double-row zinc-boxes having compartments 18 in. wide, 18 in. deep, and 34 in. long; Johnson zinc lathe; acid and vacuum filter tanks for treating precipitate; roasting furnace; and Case tilting No. 275 crucible furnace for melting precipitate.

The mill is driven by electric power supplied by the Montana Power Co. at the following rates, per kw.-hr.: 200 to 300 hp., 0.68 c.; 300 to 500 hp., 0.61 c.; 500 to 750 hp., 0.55 c.; plus \$1 per month per installed motor horsepower.

The plant as above described was operated from May, 1915, to the end of the year, when an additional Dorr thickener and a 12 by 12-ft. Portland revolving filter were added, increasing the nominal capacity of 100 tons per 24 hr.; but not the percentage of extraction. The combined treatment, at a cost of \$1.48 per ton, saved 90 per cent. of the gold and

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\*Superintendent, Gloster Mill.

59 per cent. of the silver, of which 78 per cent. was extracted by cyanide, 20 per cent. by amalgamation and 2 per cent. by concentration.

The amalgamation was accomplished in copper launders, attached to the concentrate end of the Wilfley finishing table; concentrates, at a ratio of 1 ton to 510 tons ore, averaged 12 oz. gold and 59 oz. silver per ton, and were leached with cyanide solution for 14 days to an average value of 1.1 oz. gold and 26.75 oz. silver, before being shipped to a smelter.

It will be noticed that the whole of the crushing was accomplished in two stages—gyratory crusher and Chilean mills—screen analyses of the products being given in Table 1.

TABLE 1.—*Screen Analyses of Chilean Mill Product and Classifier Products*

Mesh	Chilean Mill Product		Sands		Slimes	
	Per Cent.	Cum. Per Cent.	Per Cent.	Cum. Per Cent.	Per Cent.	Cum. Per Cent.
Over 48.....	10.9	10.9	24.2	24.2		
48-65.....	11.6	22.5	20.9	45.1		
65-100.....	17.0	39.5	29.2	74.3	2.1	2.1
100-150.....	13.6	53.1	14.0	88.3	11.1	13.2
150-200.....	7.6	60.7	5.5	93.8	12.1	25.3
Below 200.....	39.3	100.0	6.2	100.0	74.7	100.0
	100.0		100.0		100.0	

The final products were admirably adapted for the treatment of that particular ore, if the cost of all-slime treatment was to be avoided.

During this period of operation of the plant, the development of the Shannon mine was going forward, with the expectation of beginning to mill Shannon ore by July 1, 1916. The Shannon ore is different in character from that of the Piegan and Gloster mines, the grade being nearly double, with 93 per cent. of the total value in gold.

Since it was advisable to use the one mill for all the ores, an all-slime treatment was adopted, requiring the following changes and additions to existing milling equipment: A standard Dorr duplex classifier, in addition to the Akins classifier, following Chilean mills; two 5 by 16-ft. pebble tube-mills having Komata lining (each driven by 50-hp. motor, short-belt drive) with two standard Dorr duplex classifiers in closed circuit; and two 12-in. bucket elevators in the crushing department. Three of the leaching tanks were converted into thickeners (one into a simple thickener and two into single-tray thickeners); the remaining two leaching tanks were converted into Dorr agitators by increasing their depth to 20 ft; a new Dorr thickener, 29 by 7 ft., was added, and another

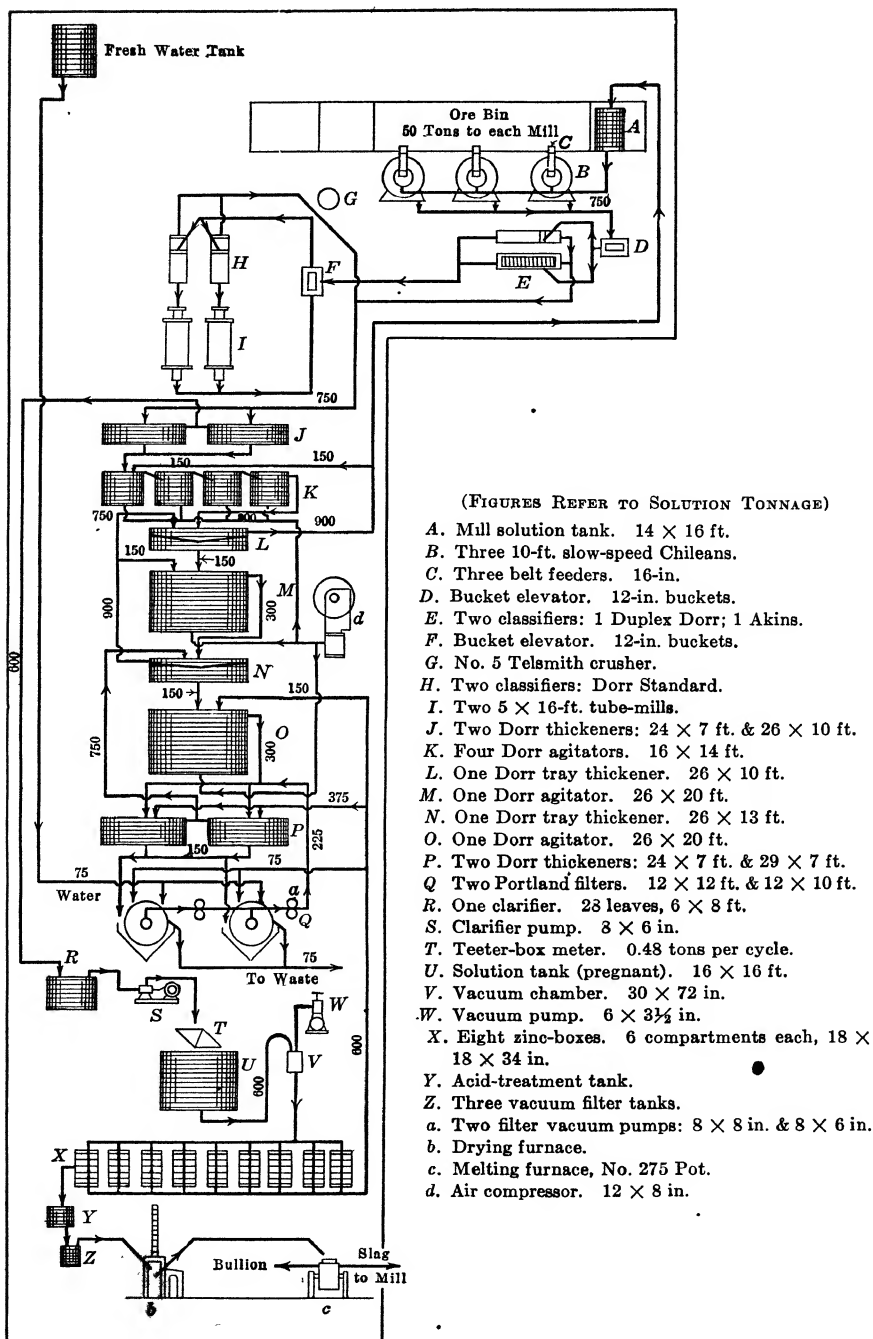


FIG. 1.—FLOW SHEET OF GLOSTER MILL.

Portland revolving filter, 12 by 10 ft., also a set of twenty-eight 6 by 8-ft. clarifying leaves. Zinc-box capacity was doubled.

The remodeled plant went into commission in August, 1916, with a nominal capacity of 150 tons per 24 hr., with 60-hr. treatment; for 10-day periods, over 200 tons per 24 hr. have been treated, with a slight decrease in extraction. Concentration and amalgamation were continued in the fine-grinding plant for a period of six weeks, and were then abandoned, with no loss in total percentage of extraction.

The unusual feature of the plant is the use of slow-speed Chilean mills for crushing the product of coarse breakers. These mills make a remarkable reduction, but leave some fine sand to be slimed in the tube-mills. Repairs are required rather frequently and access to make repairs is difficult, making the mills unpopular with the attendants. Crushing costs,

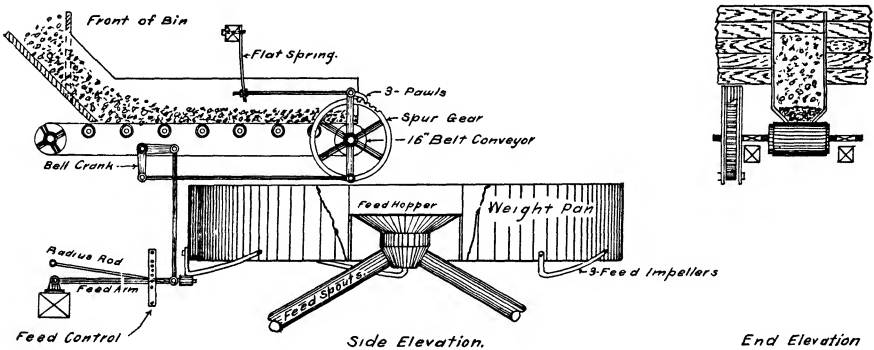


FIG. 2.—AUTOMATIC FEEDING DEVICE FOR LANE MILLS.

however, shown in Tables 4 and 5, compare favorably with other modes of coarse crushing, being, in fact, rather less than those shown by larger plants using ball-mills. An undesirable feature of these mills in a slime plant is that their product is too uniformly fine for regrinding in a pebble-mill without undue consumption of pebbles.

Treatment is now as follows (Fig. 1): crushed ore from the two mines, approximately 100 tons of Shannon and 50 tons of Gloster per day, carrying 6 and 8 per cent. moisture respectively, go into the same receiving bin, from which it is fed to the Chilean mills by belt-conveyor feeders, actuated by cams on the mills, which automatically keep a fairly uniform depth of ore between the tires and the dies (Fig. 2). To each ton of ore fed, 4.3 tons cyanide solution, 0.0375 per cent. strength, and 10 to 12 lb. of lime are added at the feed hoppers. The average rate of crushing in the Chilean mills is 2.69 tons per hour per mill, screen analysis of the feed and discharge being shown in Table 2.

TABLE 2.—*Screen Analysis of Chilean Feed and Discharge, and of Final Product*

Size	Chilean Mill Feed		Chilean Mill Discharge		Classifier Underflow to Tube-mills		Classifier Overflow	
	%	Cum. %	%	Cum. %	%	Cum. %	%	Cum. %
Over 3 in.....	0.4	0.4						
3 in.-2 in.....	2.9	3.3						
2 in.-1½ in....	7.5	10.8						
1½ in.-1 in....	17.7	28.5						
1 in. -¾ in....	43.3	71.8						
¾ in.-20 mesh..	22.9	94.7						
20-48 “	2.5	97.2	14.0	14.0	26.6	26.6		
48-65 “	(a)2.8	100.0	11.4	25.4	17.7	44.3	1.1	1.1
65-100 “	.....	.....	13.8	39.2	23.2	67.5	4.2	5.3
100-150 “	.....	.....	10.2	49.4	13.6	81.1	9.9	15.2
150-200 “	.....	.....	7.5	56.9	8.7	89.8	12.4	27.6
Below 200 “	.....	.....	43.1	100.0	10.2	100.0	72.4	100.0
	100.0		100.0		100.0		100.0	

(a) Includes all below 48 mesh.

The Chilean product is elevated to classifiers, from which the overflow goes to the slime-treatment department and sands to tube-mills; these are in closed circuit with classifiers, the overflow of which joins that

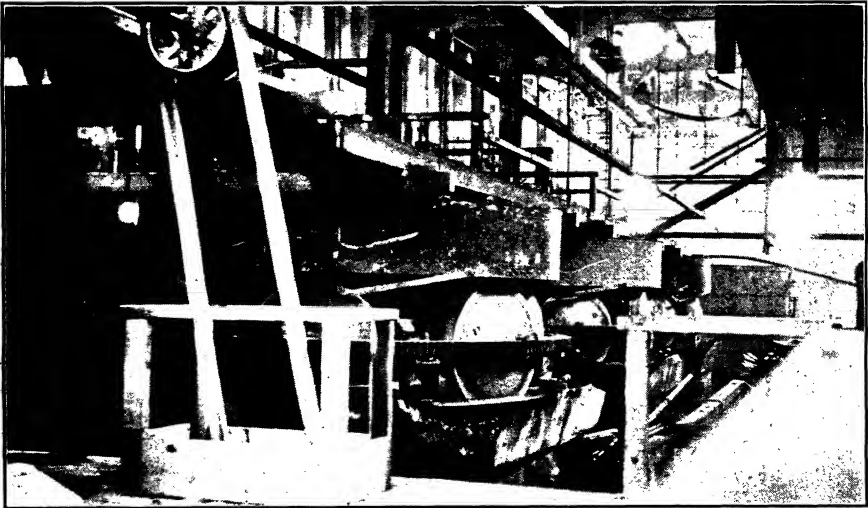


FIG. 3.—INSTALLATION OF LANE CHILEAN MILLS.

from the Chilean-mill classifiers and goes to the slime-treatment department. Table 2 gives screen analysis of the slimes as treated.

The first unit in the treatment department is composed of two Dorr thickeners in parallel, one 24 by 7 ft. and one 26 by 10 ft., the underflow, specific gravity 1.35, going to the first agitation unit composed of four 14 by 16 ft. tanks in series, where enough cyanide to increase the solution strength to 0.05 per cent. is added, and sufficient mill solution to make dilution  $2\frac{1}{2}$  to 1; the overflow goes to clarifying leaves and precipitation.

TABLE 3.—*Mill Records for 1917—Treating 52,885 Dry Tons*

	GOLD	SILVER
Average value of feed, oz. per ton. ....	0.5967	1.618
Average value of tails, oz. per ton. ....	0.0217	0.381
Recovery, per cent. ....	96.4	76.5
Combined recovery (money value), per cent. ....		94.5
Net recovery. ....		\$682,059
Ratio Au to Ag, in feed. ....		1:2.7
Ratio Au to Ag, in tails. ....		1:17.7

	TONS PER HOUR	KW.-HR. PER TON
Symons No. 5 gyratory crusher. ....	15.3	1.1
Lane Chilean mill (each). ....	2.69	7.7
Tube-mill, 5 × 16 ft. (each). ....	2.52	8.6 (a)
Thickening and agitation. ....	.....	5.8
Filtering, clarifying, precipitating, refining, and assaying. ..	..	1.5
Portland filters, 12 × 12 and 12 × 10 (both). ....	4.03	
Total. ....		24.7

(a) On basis of total mill feed; about 60 per cent. of total feed goes through tube-mills.

The second thickener unit is a 26 by 10-ft. Dorr single-tray. The underflow (specific gravity of tray product, 1.37—of tank compartment, 1.34) is raised 11 ft. net by a diaphragm pump, attached to each compartment, to the second agitation unit, which is a 26 by 20-ft. tank; here, sufficient overflow from No. 3 thickener unit is added by air lift to make the dilution  $2\frac{1}{2}$  to 1. The overflow from the second goes to mill-solution tank or to precipitation, as conditions allow.

The third thickener unit is a 26 by 13-ft. Dorr single-tray, where the underflow (specific gravity tray product, 1.35—tank, 1.30) is raised  $8\frac{1}{2}$  ft. net by a diaphragm pump attached to each compartment, to agitation unit No. 4, where sufficient barren solution is added by air lift to make the dilution  $2\frac{1}{2}$  to 1; overflow goes by gravity, counter-current, to No. 2 thickener unit.

The fourth thickener unit is composed of two tanks, 24 by 7 ft. and 29 by 7 ft., in parallel, where barren solution and filtrate from the Portland vacuum filters are mixed with pulp entering thickeners; underflow, specific gravity 1.39, is raised  $5\frac{1}{2}$  ft. net to the Portland filters, where pulp



TABLE 4.—*Costs per Ton for Milling 52,885 (Dry) Tons, 1917*

Department	Shifts Worked		Labor Costs		Material Costs		Power Costs	Total Costs
	Repairs	Operating	Repairs	Operating	Repairs	Operating		
			\$	\$	\$	\$	\$	\$
Rock breaking...	60	386½	0.0056	0.0267	0.0103	.....	0.0036	0.0462
Lane milling.....	495	1060½	0.0439	0.0814	0.0198	0.0491	0.0617	0.2559
Tube-milling.....	70¾	569	0.0073	0.0427	0.0127	0.1096	0.0702	0.2425
Chemical treatment.....	.....	70½	.....	0.0050	.....	0.3120	.....	0.3170
Agitating.....	92¾	138¾	0.0091	0.0106	0.0134	.....	0.0238	0.0569
Thickening.....	82	139½	0.0084	0.0107	0.0069	.....	0.0229	0.0489
Filtering.....	101½	1084¾	0.0090	0.0833	0.0077	0.0185	0.0061	0.1246
Clarifying.....	4	277¾	0.0004	0.0213	0.0004	0.0022	0.0029	0.0272
Precipitating.....	7	251¾	0.0006	0.0223	.....	0.1707	0.0010	0.1946
Clean-up and refining.....	4	324½	0.0004	0.0260	0.0013	0.0664	.....	0.0941
Tailings expense..	.....	626¼	.....	0.0447	.....	0.0197	.....	0.0644
Heating expense..	9½	105¼	0.0008	0.0072	.....	0.0422	.....	0.0502
Building maintenance.....	34	.....	0.0029	.....	0.0020	.....	.....	0.0049
General repairs...	134¾	.....	0.0152	.....	0.0186	.....	0.0023	0.0361
Miscellaneous expense.....	.....	344¼	.....	0.0496	.....	0.0078	0.0040	0.0614
Bullion expense...	.....	3	.....	0.0002	.....	0.0660	.....	0.0662
Oil, waste and packing.....	.....	.....	.....	.....	.....	0.0143	.....	0.0143
Assay expense....	.....	233¾	.....	0.0200	.....	0.0070	0.0032	0.0302
Totals.....	1095½	5614½	0.1036	0.4517	0.0931	0.8855	0.2017	1.7356

cake is given two washes of barren solution (one applied by a set of spray nozzles and a second by a perforated drip pipe), followed by a wash of water applied through perforated drip pipe. The filter cake, carrying 29.9 per cent. water, is then discharged as tailings and 20 per cent. more water is added to move it to the pond. The filter tailings are sampled by teeter-box sampler, operated by barren solution; the latter is also sampled by the same teeter-box. Overflow from the fourth thickener unit is pumped (counter-current) to No. 3 thickener.

The Portland filters require new filtering medium of cotton drill and burlap, and wire winding, every six to seven months; the filter is out of commission 28 to 36 hr. for removing worn wire and cloth, cleaning, applying new burlap and drill, and rewinding.

Pregnant solution going to precipitation is metered and sampled by a teeter-box holding 0.483 ton of solution per cycle; 3.81 tons of solution

and spouted on top of the tank charge in the agitation circle. Above the separation zone, the water or solution rises clear within the diaphragm, and by the hydrostatic pressure of the pulp undergoing agitation the clear solution is raised to a height above the level of the pulp commensurate with the depth of the sides of the diaphragm.

As an example, if the pulp being agitated contained 2 parts by weight of solution to 1 part by weight of solids, it would have the specific gravity of, say, 1.26, (depending on the specific gravity of the solids) or, about one and one-quarter times the weight of the same volume of water. Therefore, if the sides of the diaphragm extend, for example, 9 or 10 ft. down into the pulp, the weight of the pulp would raise and balance a

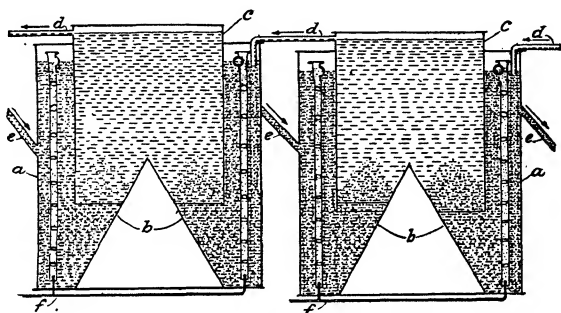


FIG. 2.

FIG. 3.

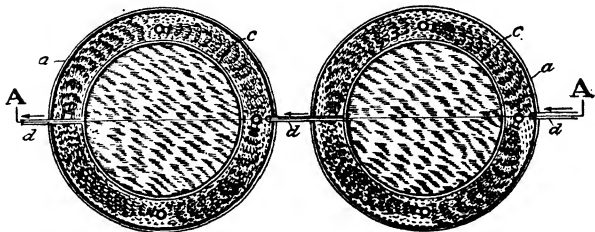


FIG. 4.

FIG. 5.

column of clear solution within the diaphragm to a height of more than 2 ft. above the level of pulp in the agitation circle. The separation of the solution from the solids takes place rapidly in the separation zone at the foot of the diaphragm, probably due in a considerable degree to the centrifugal force set up by the rotary flow of the pulp in the agitation circle. At any rate, clear solution separates from the pulp mass in this region and rises rapidly and continuously within the diaphragm from the top of which it is taken off through pipes *d* and delivered into the pulp being agitated in the next adjoining tank, and so on until from the head tank of the series the solution goes to the clarifiers to remove any fine slime that may be carried over in suspension, and thence to precipitation.

The series of tanks in which the treatment is carried on is set so that each tank from head to tail of the series is 6 in. below the level of the tank preceding it. This height is quite sufficient to overcome the flow friction of the pulp as it goes from tank to tank through pipes *e*. The last two tanks of the series may be specially arranged so that the pulp may be thickened to a very high density before it is discharged to filter press or dump as conditions require. In this way, the counter migration of the solids and solution is brought about automatically and continuously through any number of tanks equipped as shown in the illustrations. The precipitated cyanide solution, which is added to the last tank of the series, flows from tank to tank toward the head of the series, dissolving the metals from the solids with which it is agitated as it goes. The solution thus becomes enriched as it goes from tank to tank until it flows from the head tank for precipitation, while the solids being carried from the head to the tail tank of the series become poorer and poorer until they are discharged barren from the tail tank.

The same principle may be used in the acid leaching of copper ores, but the tanks and the transfer pipes used for this purpose are made of wood in order to be acid proof. This method obviates the use of pumps between the tanks, which give great trouble in handling acid solution.

The agitation of the pulp may be carried on within the diaphragm, leaving the annular space outside the diaphragm for the rise of the clear solution. I have designed this latter method for a plant having Pachuca tanks, but the first described method may also be installed in Pachuca tanks to advantage because the clear solution may be lifted to any height which allows a series of tanks set on a level to be operated without the intervention of pumps.

Some time ago I suggested to the operator of a plant of Pachuca tanks that he equip the tanks with the apparatus I have described, but he was skeptical that water could be made to rise above its own level so as to flow from tank to tank as I described.

A 12-ft. length of stack was available and this was suspended in the tank to a depth of 10 ft. below the level of the pulp being agitated. In 2 hr. clear solution was overflowing the top of the pipe, which was 2 ft. above the pulp level, a continuous overflow as from an artesian well.

In the hydrometallurgical treatment of ore pulps, the principle developed as described and applied will be found to be an important forward step. The cost of installation is small and the cost of operation is nothing.

## Effect of Oxygen on Precipitation of Metals from Cyanide Solutions

BY THOMAS B. CROWE,\* E. J. M., VICTOR, COLO.

(Colorado Meeting, September, 1918)

MUCH has been written upon the precipitation of metals from cyanide solution by zinc. We often read of the many factors that influence precipitation, such as zinc surface, purity of zinc, percentage of lead, temperature of solutions, strength of solutions in cyanide or alkali, etc. Little do we hear, however, of the part that oxygen plays in precipitation.

Caldecott<sup>1</sup> says: "As the dissolving of gold is essentially an oxidation process, so its precipitation is one of reduction." All who have studied or worked with cyanide solutions will agree that Caldecott's statement is true. In the cyanide work in the mills of the Portland Gold Mining Co., we have always found oxygen to be the greatest enemy to precipitation.

### OXYGEN ANTAGONISTIC TO GOOD PRECIPITATION

A few years ago, while making some experiments on the effect of pressure upon the dissolving rate of gold from our ores, to prove a point, I made some bottle tests under vacuum. The results were surprising, and showed that under vacuum practically no gold dissolved, which confirmed my belief that Elsner's equation was true and also led me to investigate the amount of dissolved air carried by solutions at atmospheric pressure.

The experiment consisted in partly filling an acid-bottle with mill-solution and connecting it by a rubber hose to a vacuum pump. When the vacuum was suddenly applied, a cloud composed of thousands of small air bubbles rose out of the solution, showing qualitatively the amount of dissolved air that was contained in the solution, and also proving that the relief of atmospheric pressure on the solution permitted this dissolved air to escape.

In studying literature on the subject, we find Henry's law—"The amount of gas dissolved by a liquid is proportional to the pressure to which the gas is subjected."

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\* Mill Superintendent, Portland Gold Mining Co.

<sup>1</sup> W. A. Caldecott: *Chemistry of Banket Ore Treatment*, 389; in "A Text-book of Rand Metallurgical Practice" by Ralph Stokes and others. London, Charles Griffin & Co., Ltd., 1912.

We find that at atmospheric pressure water dissolves from 2 to 4 per cent. of air by volume.

We also find that when air is dissolved by water, owing to the different coefficients of absorption of oxygen and nitrogen, the absorbed air has the composition of about 35 per cent. oxygen and 65 per cent. nitrogen, while ordinary atmospheric air is about 21 per cent. oxygen and 79 per cent. nitrogen. Mill solutions, then, contain not atmospheric air, but a per-oxidized atmospheric air, which is very good for oxidizing but very injurious to the reduction process of precipitation.

In the early days of zinc-dust precipitation, the dust was sprinkled over the top of a tank of solution while under agitation; large quantities of zinc were used and the solution tailing was far from what was desired.

Later Merrill's improved method came out, the principal characteristic of which was the exclusion of air from contact with the precipitant and precipitate during precipitation. Merrill's method was a big improvement over the old scheme, allowing a material reduction in the quantity of zinc used, with much lower barren solutions, and showed very plainly the effect of oxygen in former work.

Having had the sad experience of allowing free air to pass with my solution through the precipitation press, and knowing that Merrill's process had been an improvement over the old open-tank process, I naturally reasoned that the removal of the dissolved air from solution would be the means of further benefiting precipitation, providing a vacuum could be practically applied to solution, prior to, or during precipitation.

Laboratory tests on the application of a vacuum to solution before the addition of zinc-dust confirmed my theory. Large tests were tried, apparatus was designed that would continuously apply a vacuum to solution during its flow to the precipitation presses, and gradually the vacuum system of precipitation worked its way into our mills, with the result that the zinc consumption has been cut in half, which fact is especially gratifying to us during the war period with its prevailing high prices.

#### EFFECT OF VACUUM PRECIPITATION ON CONSUMPTION OF CYANIDE

Having been successful in reducing the amount of zinc used, and as the price of cyanide kept soaring, we decided to try to make a reduction in cyanide consumption.

A thorough scrutiny of our former work revealed the fact that, for dissolution purposes on our particular ore, the cyanide solution strength was much higher than was necessary, having been kept high for the sake of good precipitation. In fact, the solution strengths in our mill had been reversed—the strongest solution was the barren; and this being used as a wash on the filters, occasioned high cyanide consumption. The

high strength of the barren solution was caused by the necessity of adding lump cyanide at the head of the precipitation process to insure low-grade solutions to be used in washing.

With the installation of our vacuum system, we found that perfect precipitation could be maintained without the addition of lump cyanide, and today our mills are running with the highest solution strength in the agitators, and not in the barren tank. This, together with a generally lower solution strength in all parts of the mill, has allowed a material reduction of cyanide consumption.

Table 1 will give an idea of the amount of cyanide and zinc used before and after the installation of the vacuum precipitation.

TABLE 1.—*Comparison of Ordinary and Vacuum Precipitation Systems*

	TONS OF SOLUTION PRECIPITATED	POUNDS NACN PER TON ORE	POUNDS ZN PER TON ORE	POUNDS ZN PER TON SOL.
12 months, Year 1915.....	503,819	0.331	0 407	0.174
12 months, Year 1916.....	536,455	0.320	0 369	0.150
6 months, Year 1917.....	249,988	0.175	0.191	0.084

Years 1915 and 1916, ordinary zinc-dust precipitation. Year 1917, vacuum precipitation.

#### OPPOSING ACTION OF OXYGEN AND HYDROGEN

How often have we found, when working with zinc shavings, that the only remedy for poor precipitation, excepting the addition of lump cyanide to the head of the box (which has its limitations), is a cleanup or redressing of the box.

Assays of the effluents from the different compartments of a box during a period of good precipitation generally reveal the fact that each compartment in the series is doing some work, there being a gradual reduction of the value of the solution as it progresses from compartment to compartment.

A similar series of assays taken during a period when precipitation is only fair, the box being inclined to give erratic results, shows that the first compartments of the series are doing very little work, the burden consequently being thrown on those compartments lower in the series. A third series of assays, taken when precipitation is bad, generally shows that not only is no precipitation occurring in the first compartments, but that the first compartment is giving up its gold, the values being carried to the lower compartments and sometimes entirely out of the box. Decreasing the rate of flow of solution through the box sometimes helps this condition and increasing the cyanide strength also gives temporary relief, but a redressing of the box seems to be the only real remedy.

During precipitation, two opposing forces are continually working: first, a precipitating force; second, a dissolving force. The precipitating

force is a reducing action and is caused by the dissolving of the zinc, which creates an electro-motive force and evolves hydrogen. Nascent hydrogen seems to be the active agent that causes precipitation. The second, or dissolving force, is an oxidizing action, its strength being a function of the amount of oxygen carried by the solution.

Reducing agent hydrogen and oxidizing agent oxygen are decided enemies, and cannot live together in the same solution. When nascent hydrogen, caused by the dissolving of zinc in cyanide solution, is generated, its first work is that of combating its enemy, oxygen, combining with it and getting it out of the way. After this is done, it then takes up the role of precipitation and plays its part.

When large quantities of zinc are dissolved and plenty of hydrogen is evolved, as is the case when lump cyanide is added to the head of the box, the effect of oxygen becomes insignificant, but when the cyanide solution strength is low and very little zinc is dissolved, then the oxygen in solution may gain supremacy over the evolved hydrogen and result in poor precipitation or, in extreme cases, dissolution of precipitated gold. Especially is this true when the zinc has been in use for long periods and the first compartments are rich in gold and poor in zinc. A freshly dressed box presents more zinc surface and, in a cyanide solution of a given strength, has a certain power to reduce and precipitate, this power becoming less and less as it does its work. This work consists of a very small job of precipitating and a very large job of counteracting the effects of oxygen contained in the air dissolved in solution.

Why then allow this enemy of precipitation to pass with the solution into the zinc press or box, when it can so easily be removed by the application of a vacuum during its flow?

When the air in solution is removed prior to precipitation and the deoxidized solution is run or pumped through the precipitating system, only the burden of precipitating is put upon the zinc and we find, when this is done, that a very weak electro-motive force is sufficient to effect precipitation, which means that one can operate with both weaker cyanide-solution strength and with smaller amounts of zinc, producing a higher-grade precipitate with less cost for cutting down and refining.

## DISCUSSION

G. H. CLEVINGER, Colorado Springs, Colo.—Mr. Crowe's paper will be of great interest to cyanide operators, as it is a distinct new development in cyanidation. After reading the paper it occurred to me that some of us had done practically the same thing some time ago, but without design and unconsciously. I refer particularly to the operation of some of the earlier types of vacuum filter, used in connection with a dry vacuum pump and a receiver, which is somewhat similar equipment to

that employed by Mr. Crowe. However, we did not appreciate the good effects we were getting from such of this solution as was precipitated, and it has remained for Mr. Crowe to bring distinctly to our attention the advantages of removing air from cyanide solutions before precipitation. Full credit is therefore due him for this very clever improvement in the cyanide process.

J. V. N. DORR, New York, N. Y.—I think that Mr. Crowe's work has been the greatest single improvement on the chemical side of cyanidation within the past 15 years; other improvements within that time have been largely mechanical. Although he has shown us something that each of us should have known before, I feel that he has made a very definite and valuable contribution to the art.

A. L. BLOMFIELD, Colorado Springs, Colo.—When we first tried Mr. Crowe's process at the Golden Cycle mill, we supposed that the precipitation boxes would have to be kept covered, lest the air should again be dissolved in the solution and disturb the precipitation; however, we found that nothing that we did made the slightest difference. The application of the system had the immediate effect of doubling the capacity of the boxes and giving about 30 per cent. better tailings.

G. T. HANSEN,\* Salt Lake City, Utah.—We have had occasion, within recent months, to heat pregnant solution to about 170° F., for the purpose of precipitating copper, before sending it to the zinc boxes. We found that heating accomplishes practically the same result, as regards removal of air, as applying a vacuum.

GEORGE M. TAYLOR,† Colorado Springs, Colo.—I would like to say, on behalf of Mr. Crowe, that at our two mills, the Victor and the Independence, where we were treating about 2000 tons of ore per day, there was a saving in zinc and cyanide of about \$2500 a month, or \$30,000 a year.

LOUIS D. MILLS, San Francisco, Cal. (written discussion‡).—The principle involved in the Crowe vacuum precipitation process is so elementary and the apparatus required is so simple, that the whole subject affords scant material for a technical discussion. As one prominent Colorado metallurgist recently remarked when asked to contribute to this paper: "The Crowe process is the biggest step forward since the early days of the cyanide process, but there is nothing to write about; just put it in your mill and try it." The writer confesses to a similar

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\* Manager, Midvale Minerals Co.; Dayton Placer Recovery Corp'n.

† Manager, Milling Department, Portland Gold Mining Co.

‡ Received Nov. 12, 1918.



feeling, but having been closely identified with the process since its inception in 1916, he is perhaps in a position to at least comment on the theory involved and to record some of the results that have been achieved in actual practise.

In practising the Crowe process, the solutions to be precipitated are first vacuumized in a suitable dispersion tower; that is, thin films of the liquid are subject to the action of a vacuum within a receiver. Substantially all of the dissolved air is thus removed through the vacuum pump, and the treated solution is sent to precipitation without further opportunity for re-absorption of air. The time of contact required is short and the amount of power consumed is very small. A receiver 6 ft. (1.8 m.) in diameter by 12 ft. long handles upward of 3000 tons of solution in 24 hr. or 500 gal. (1892 l.) per min., with a consumption of under 1 hp. No supervision is required other than to insure the proper operation of the small vacuum pump and motor, the flow of solution being entirely automatic.

The actual precipitation of the metals may be accomplished by any of the well-known methods, as, for instance, in zinc shavings boxes or by the use of powdered precipitants, such as zinc, aluminum, or charcoal, as in the Merrill process. The writer agrees with Mr. Crowe, that the true precipitating or reducing agent is invariably nascent hydrogen. This hydrogen is probably evolved first by the dissolution of the precipitant in caustic alkali and the reaction continues as long as fresh precipitant is exposed to the solvent. A portion of the hydrogen evolved by this dissolution is lost, as far as the operation of precipitation is concerned, by combining with the oxygen of the dissolved air contained in all cyanide solutions.

The amount of dissolved oxygen in such solutions depends upon the pressure and agitation to which the solutions have been subjected. Contact with reducing minerals of the ore tends to rob the solution of its oxygen, but, to insure maximum dissolution of the precious metals, the oxygen content is kept as near as possible to the saturation point. The solubility of oxygen in cyanide solutions varies inversely with the temperature, being relatively much greater in cold solutions than in warm solutions. This explains the well-recognized advantages of warming cyanide solutions during cold weather.

The oxygen contents of normal treatment solutions varies from 4 to 8 mg. per liter, and 6 mg. may be taken as an average figure.<sup>1</sup> The usual equations show that 6 mg. of oxygen per liter are equivalent to<sup>2</sup>

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<sup>1</sup> W. A. Caldecott: Chemistry of Banket Ore Treatment, in "A Text Book of Rand Metallurgical Practice," by Ralph Stokes and Others. 1, 385. London, 1912, C. Griffin & Co., Ltd.

<sup>2</sup> H. A. White: The Estimation of Oxygen in Working Cyanide Solutions. *Journal, Chemical, Metallurgical and Mining Society S. A.* (June, 1918) 18, No. 12, 296.

- 0.012 lb. of oxygen per ton of solution.
- 0.049 lb. of zinc per ton of solution.
- 0.195 lb. of potassium cyanide per ton of solution.
- 0.150 lb. of sodium cyanide per ton of solution.

Thus it is evident that in precipitating a solution containing in excess of 0.20 lb. KCy or 0.15 lb. NaCy sufficient zinc must be wastefully dissolved to satisfy the dissolved oxygen present, the ultimate product being the double salt  $\text{Na}_2 \text{Zn Cy}_4$ , and the quantities entering into the reaction being 0.012 lb. of oxygen, 0.049 lb. of zinc and 0.150 lb. of sodium cyanide, respectively. The removal of this oxygen prior to precipitation should therefore reduce the consumptions of zinc and cyanide by the foregoing amounts, and it is of interest to note that this exact result has been found in the application of the Crowe process.

At the Belmont mill in Tonopah the chemical consumptions prior to the installation of the vacuum process were as follows: Zn, 0.772 oz. per fine ounce of silver recovered; NaCy, 2.850 lb. per ton ore milled. After installing the vacuum process, the figures became: Zn, 0.650 oz. per fine ounce silver recovered, which corresponds to a saving of 0.14 lb. per ton ore milled; at the same time the cyanide consumption was reduced to NaCy, 2.40 lb. per ton ore milled, or a saving of 0.45 lb. per ton ore milled.

Since at this plant the ratio of solution precipitated to ore milled is very close to 3:1, the actual reductions per ton of solution precipitated, namely 0.046 Zn and 0.15 NaCy, are practically identical with the theoretical figures.

Considered from an economic standpoint and remembering that zinc and sodium cyanide are at present worth approximately \$0.15 per lb. and \$0.30 per lb., respectively, the foregoing figures furnish a convincing illustration of the value of the Crowe process, the saving in chemicals alone being in excess of \$0.15 per ton of ore milled.

## Roasting for Amalgamating and Cyaniding Cripple Creek Sulfo-telluride Gold Ores

BY A. L. BLOMFIELD,\* B. E., AND M. J. TROTT,† E. MET., COLORADO SPRINGS, COLO.  
(Colorado Meeting, September, 1918)

THE Golden Cycle Mining and Reduction Co. operates its custom mill at Colorado Springs on Cripple Creek ores exclusively. These ores are straight sulfo-tellurides, with practically no base metals such as Pb, Cu, Zn, As, or Sb.

Upon entering the mill, the ore all passes through the sampler to storage bins, where beds of 5000 tons each are made to insure uniformity in roasting.

### CLASSIFICATION OF ORES

For efficiency, it has been found advisable to separate the ores into two classes according to their lime content. Other constituents have their effect, such as alumina and magnesia, but, speaking generally, these are roughly proportional to the lime, which, as CaO, is used as the indicator. Class A contains not more than 2 per cent. CaO and comprises about 66 per cent. of the ore treated. Class B contains over 2 per cent. CaO, and comprises 34 per cent. of the ore treated. Typical analyses of both classes are as follows:

	CLASS A	CLASS B
Insol.....	86.70	75.90
Al <sub>2</sub> O <sub>3</sub> .....	2.30	3.40
Fe.....	3.26	3.48
CaO.....	1.57	5.12
S.....	1.79	1.80
MgO.....	0.21	1.08
Ignition loss.....	3.20	6.50
	<hr/>	<hr/>
	99.03	97.28

The beds of each class of ore are made as large as possible with the available ore as it comes in, to avoid changing from one bed to the other any oftener than necessary. Each roaster will handle 50 per cent. more of class A ore than of class B, with practically the same results. This necessitates changing the rate of feed for each new bed and tends toward a poor roast and loss of tonnage at each change.

\* Manager, Golden Cycle Mining & Reduction Co.

† Assistant Superintendent, Golden Cycle Mining & Reduction Co.

## CRUSHING PREPARATORY TO ROASTING

After leaving the storage bins, the ore is crushed in Schmidt "kominuters," ball-mills. Diagonal slotted screens  $\frac{9}{64}$  by  $\frac{1}{2}$  in. (3.57 by 12.7 mm.) are used, giving a product which analyzes as shown in Table 1.

TABLE 1.—*Screen Analysis of Ball-mill Product*

Size	Screens		New Screens	
	Per Cent.	Cum. Per Cent.	Per Cent.	Cum. Per Cent.
Over $\frac{5}{32}$ in. . . . .	6.1	6.1	1.1	1.1
$\frac{5}{32}$ + $\frac{1}{8}$ in. . . . .	5.1	11.2	2.4	3.5
$\frac{1}{8}$ in.—10 mesh. . . . .	24.0	35.2	16.6	20.1
10—20 mesh. . . . .	22.2	57.4	26.2	46.3
20—30 mesh. . . . .	8.0	65.4	10.2	56.5
30—40 mesh. . . . .	5.2	70.6	7.6	64.1
40—60 mesh. . . . .	6.4	77.0	9.8	73.9
60—80 mesh. . . . .	1.2	78.2	1.2	75.1
80—100 mesh. . . . .	2.5	80.7	3.0	78.1
100—150 mesh. . . . .	2.2	82.9	3.9	82.0
150—200 mesh. . . . .	4.2	87.1	4.4	86.4
Below 200 mesh . . . . .	12.9	100.0	13.6	100.0
	100.0	.....	100.0	

Crushing efficiency is increased by coarse crushing in "kominuters," both in "kominuters" themselves and in Chilean mills after roasting.

The finer the crushing, the greater the dust losses in the roasters. If too fine, there is a tendency for the hot ore to "run" through the roasters.

The coarser the crushing, the harder it is to roast the mechanically occluded sulfides. The ore may appear to be well roasted when too coarse, but, after regrinding, will fill the solutions with soluble sulfides, which increase the consumption of cyanide. The coarse ore itself does not seem to entail a high residue, but the bad effect upon the solution tends to hinder extraction, unless the plant is very flexible.

The tendency for general efficiency is toward finer crushing for the more basic ores than for the siliceous. This is roughly adjusted by finer screening before the ore enters the "kominuters" and by using the mills with the newest screens. The average should be not more than 30 per cent. coarser than 10 mesh.

## ROASTING FURNACES

The roasting furnaces used are the Edwards Duplex, 54 rabble, supplied by Stearns-Rogers Mfg. Co. The length is 115 ft. (35 m.); width, 13 ft. (3.9 m.); live roasting area, 1495 sq. ft. (138.8 sq. m.); coolers, 44 ft. (13.4 m.) long and 13 ft. (3.9 m.) wide, with an area of

572 sq. ft. (50.4 sq. m.). There are three fire boxes for each roaster. The temperature of the ore at the discharge point of the roasters is about 485° C.; at the discharge point of the coolers, about 278° C. The temperature depends on tonnage, air temperature and the rate of rabbling. Trials were made on rabble speeds from 1½ r.p.m. up, and the most efficient cooling was at a rabble speed of 6 r.p.m.

### *Details of Rabbles*

The spindles, rabbles and teeth are made of cast iron by a local foundry. The consumption of teeth is 0.1637 lb. per ton of ore; of rabble arms, 0.0684 lb. per ton of ore, and of spindles, 0.0265 lb. per ton of ore.

Inside the roaster, all rabble arms are water-cooled, the water being pumped from Fountain Creek, just below the mill, to a storage tank on the hill above the roasters. After passing through the rabbles, it is run over a cooling tower and pumped back to the storage tank. The temperature of the water going into rabbles is 25° C. to 34° C., depending upon the temperature of air, the wind, and the tonnage in roasters. Its temperature on leaving the rabbles varies from about 45° C. to 64° C. The water used per roaster per minute varies from 90 to 115 gal. depending upon the condition of the rabbles and the rate of feed.

There are 27 pairs of rabbles in each roaster and 11 pairs in the cooler. In the roasters they travel 3 r.p.m. and in the coolers, 6 r.p.m.

The hearth is sloped from feed to discharge end, ½ in. to the foot (4.17 cm. to the meter), which is sufficient to cause the ore to move forward under the action of the revolving rabbles. For a given rate of feed, the slower the rabble speed the greater the depth of ore. The greater the rabble speed, the less tendency there is to burn or "ball" the ore under excessive heat.

The average time of passage of the ore through the roasters is 6 hr. From 13 to 17 hp. is required for each roaster and cooler, depending on the feed and the speed of the rabbles.

The roasted ore falls from the cooling hearth through a choke feeder onto a 315-ft. (96 m.) reciprocating drag conveyor. The sheet-iron fins are 34 in. (86.36 cm.) long by 8 in. (20.32 cm.) deep. An average of 16¼ hp. is required to operate it.

The roasters are shut down the first of every month to allow repairs to be made on this drag. The pins that hold the fins are subject to great wear near the discharge end where water is sprayed on the ore before it falls on a rubber conveying belt. This belt is made of specially vulcanized rubber (Diamond Rubber Co.) to withstand the heat and moisture. The ore is at about 90° C. when it falls on this belt and contains 2 per cent. moisture. The contained heat serves, unaided, to warm the cyanide building throughout the year. It has, also, the un-

fortunate effect of warming the cyanide solution in which the ore is crushed, thus putting large quantities of calcium sulfate into solution.

### *Coal Used for Roasting*

The coal is Colorado Springs lignite, supplied by the Pikes Peak Cons. Fuel Co. from their mines 6 miles north of the city. The average analysis is as follows: Water, 20.2; volatile, 54.65; fixed carbon, 19.75; ash, 5.1; sulfur, 0.30; average heating capacity, as mined, 8700 B.t.u. For class A ore, 249 lb. of coal per ton of ore is required for roasting, while for class B ore, 347 lb. is needed. The fire-box is that of the Western Fire-Box Co., a semi-gas producer, requiring the use of live steam; 12 per cent. additional coal is used to generate this steam. The capacity per roaster ranges, for class A ores, from 125 to 150 tons per 24 hr., and for class B ores, from 80 to 100 tons per 24 hours.

### *Flue Losses*

Extensive stack tests were made in 1909 to determine the flue losses. The method was the use of the Pitot tube for measuring velocities and employing bags for collecting the dust. The volume of flue gases per roaster was 15,000 cu. ft. per min. At the time of the first tests, the revolving cooler was in use and the stack loss amounted to 13.2 c. per ton on 1-oz. ore. After changing to the present coolers, the dust loss was 3 to 4 c. per ton of 1-oz. ore. The amount of flue-dust collected was formerly from 0.7 to 0.9 per cent. by weight and carried a value 32 per cent. higher than the ore. It is now 0.4 per cent. by weight and carries a value 20 per cent. higher than the ore. The open cooler and more careful feeding and discharging has made this difference.

### *Conditions of Roasting*

The temperature, as indicated by Brown indicating pyrometers, is as follows:

Class A ore—800° to 870° C. in the center of the arch, 8 ft. above No. 1 fire-box and in the flow of heat; 800° to 850° C. 8 ft. above No. 2 fire-box.

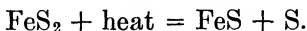
Class B ore—850° to 900° C. above No. 1 fire-box; 850° to 870° C. above No. 2 fire-box.

In roasting all ores, No. 3 fire-box keeps the ore at a red heat up to the point of discharge. The hotter the ore is kept here, the less tendency there is for the sand of class B ore to "set" in the leaching tanks. Class A ores show no such tendency within the present treatment conditions. Nevertheless, they will do so under other conditions of classification and filling.

A few practical points in the operation of the roasters are worthy of notice. The draft should be kept up to about 0.2 in. of water at all times to allow a free escape of the gases and consequently a more oxidizing atmosphere. The flames from the fire-boxes must be given an excess of air to keep them from being smoky, as their reducing effect then is quite strong.

### CHEMICAL REACTIONS IN ROASTING

#### *Siliceous Ores*



FeO may further oxidize to  $\text{Fe}_2\text{O}_3$ .

$\text{FeSO}_4$  is found when roasting at low temperatures, but at high temperatures this decomposes into FeO and  $\text{SO}_3$  or  $\text{SO}_2 + \text{O}$ .

#### *Basic Ores*

In the roasting of calcareous ores containing metallic sulfides, if sufficient sulfur be present the lime is converted almost quantitatively into  $\text{CaSO}_4$ . The behavior of  $\text{CaSO}_4$ , when heated in the presence of CO and C, has been investigated by H. O. Hofman and W. Mostowitsch.<sup>1</sup> The chemical reactions and temperatures stated herein are quite largely taken from their reports. They are given, as they appear to fit the class B ores as roasted.

The reduction of  $\text{CaSO}_4$  by CO is indicated in the equation  $\text{CaSO}_4 + 4\text{CO} = \text{CaS} + 4\text{CO}_2$ . The reaction takes place without any loss of sulfur. Very little reaction takes place unless the temperature is above  $680^\circ \text{C}$ . Between  $750^\circ$  and  $850^\circ \text{C}$ . the action is quite rapid and is completed at  $900^\circ \text{C}$ .

The reduction of  $\text{CaSO}_4$  by carbon is indicated in the equations  $\text{CaSO}_4 + 4\text{C} = \text{CaS} + 4\text{CO}$ , and  $\text{CaSO}_4 + 2\text{C} = \text{CaS} + 2\text{CO}_2$ . The action begins fairly well at  $700^\circ \text{C}$ . and finishes at  $1000^\circ \text{C}$ ., proceeding most rapidly between  $800^\circ$  and  $900^\circ \text{C}$ . At about  $800^\circ \text{C}$ .,  $\text{CaSO}_4 + 2\text{C} = \text{CaS} + 2\text{CO}_2$ ; above  $800^\circ \text{C}$ .,  $\text{CaSO}_4 + 4\text{C} = \text{CaS} + 4\text{CO}$ .

It was found that CO acted more readily and effectively than carbon alone, both beginning and finishing at lower temperatures. It is probable that the gases  $\text{CH}_4$ ,  $\text{C}_2\text{H}_6$ , and H would act similarly in the reduction of  $\text{CaSO}_4$ , since they are effective with  $\text{Na}_2\text{SO}_4$ .

CaS, if roasted in pure, dry air, does not all revert to  $\text{CaSO}_4$ , but forms a product of about 76 per cent.  $\text{CaSO}_4$  and 24 per cent. CaO, with a loss of approximately 32 per cent. S. This loss of S is accounted for by the interaction of  $\text{CaSO}_4$  and CaS, according to the reaction  $3\text{CaSO}_4 + \text{CaS} = 4\text{CaO} + 4\text{SO}_2$ .

<sup>1</sup> *Trans.* (1910), 41, 763.

The complete elimination of sulfur at this point is probably prevented by the failure of intimate contact between the  $\text{CaS}$  and  $\text{CaSO}_4$ . Action between  $\text{CaSO}_4$  and  $\text{CaS}$  takes place in a neutral or oxidizing atmosphere, but not in a reducing one.

When heating pure  $\text{CaSO}_4$  in the presence of oxygen it loses its combined water at  $900^\circ \text{C}$ . and begins to decompose at  $1250^\circ$  to  $1300^\circ \text{C}$ . In the presence of  $\text{SiO}_2$  it starts to decompose and gives off  $\text{SO}_2$  at  $1000^\circ \text{C}$ . and decomposition is completed at  $1250^\circ$  to  $1300^\circ \text{C}$ . The action is shown in the equation  $\text{CaSO}_4 + \text{SiO}_2 = \text{CaSiO}_3 + \text{SO}_2 + \text{O}$ .

An interesting equation is  $\text{CaS} + \text{FeSO}_4 = \text{CaSO}_4 + \text{FeS}$ . It takes place at a low temperature.

#### ROASTING FOR CYANIDE TREATMENT

In roasting for cyanide treatment, both sulfates and soluble sulfides are deleterious. Soluble sulfides are active cyanicides and attack zinc freely. They are active reducing agents and under certain conditions can completely eliminate all oxygen from the solutions.

Though precipitating gold to an appreciable extent only in acid solution, when the solution percolating a leaching charge becomes deoxidized by an excess of soluble sulfide, either gold is precipitated or the undissolved gold is so coated as to render further dissolution so extremely slow that to obtain a commercially good residue is impossible. In agitating slimes, especially without air, a similar condition is brought about.

It is interesting to note that if such a residue is completely dried or thoroughly treated with a solution of permanganate of potash or other effective soluble oxidizer, then subjected to cyanide treatment, the normal residue is obtained rapidly.

Acid sulfates act as virulent cyanicides if not quickly neutralized. Calcium sulfate, formed either in the roasters or by reaction of soluble sulfates in the ore on meeting the lime in solution, is decidedly deleterious. The hot ore warms the crushing solution. This becomes saturated with calcium sulfate which precipitates as the solution cools. These crystals fill pipes, launders and Dorr thickeners, render filter mats and clarifying mats impermeable, coat Merrill filters, frames and cloths and form a deposit on the zinc and zinc boxes. In class A ores these troubles are comparatively unimportant, although considerable lime is required to neutralize the solutions, whereas class B ores often require none.

#### *Formation of Soluble Calcium Sulfate*

We believe that the chief formation of soluble calcium sulfate, with class B ores, occurs in the roasters. It will be noted in the roaster equations quoted that, somewhere above  $870^\circ \text{C}$ ., the final molecule of water



is driven out of the calcium sulfate; it then becomes practically insoluble in working solutions and does not form a cement in the sand tanks.

For illustration, a ton of deposited calcium sulfate crystals and slime, if fed slowly or rapidly into the Chilean mills, forms a band of cement sand in the leaching vats and redeposits throughout the plant. If, however, 10 tons of this material is fed into the "kominuters" and thence through the roasters, meeting a heat of 870° C. or higher, it cannot be noticed in the cyanide plant.

It is commercially possible to roast ores so that they may be treated with the ease of a free-cyaniding raw ore; this is what we call an "over-roast." Such ore can be extracted to a finished residue in a good standard cyanide plant. Chemical consumption is held at a minimum. Standard classification is sufficient and no change of solution is necessary. Dissolution is rapid and reaches a definite end point. But the roast is decidedly more expensive, and the final residue much richer than that now obtained.

Table 2 shows the value of typical sand residues from class B ore, first that from an over-roasted ore, and second, an average sand residue such as is now obtained.

TABLE 2.—*Value of Sand Residues from Class B Ore*

Screen Mesh	Over-roasted Ore		Average Residue	
	Weight, Per Cent.	Gold, Ounce	Weight, Per Cent.	Gold, Ounce
Over 20.....	....	.....	1	0 01
20-30.....	4	0.03	5	0 01
30-40.....	14	0.04	15	0 01
40-60.....	23	0 05	35	0.02
60-80.....	14	0.04	} 16	0 02
80-100.....	16	0.05		
100-150.....	11	0.06	12	0 02
150-200.....	10	0.09	13	0 02
Below 200.....	8	0.12	3	0 04
	100	0.0571	100	0.0185

The slime residues show even a greater difference in gold content due to over-roasting, the difference being more marked in class B than class A ores.

Having found that a better residue could be obtained by a cheaper and, as indicated by ordinary sulfur analyses, a less complete roast, it was decided to attempt a more efficient treatment by more delicate roasting. This required a quick method of chemical testing and the

use of pyrometers to help regulate the temperature of each roaster continuously.

Chemical tests were employed for governing the roast. For class A ores a simple analysis of total, soluble, and insoluble sulfur is a sufficient guide to an experienced fireman, which, with the temperature and appearance of the roaster, soon enables him to regulate his fires. The feed is changed only by the foremen.

## RAPID METHODS OF ANALYSIS FOR SULFUR IN ORES

### *Total Sulfur in Roasted Ore*

Weigh out 1.373 gm. ore, add about 11 drops of a saturated solution of  $\text{KClO}_3$  in  $\text{HNO}_3$ , and two or three drops of  $\text{HCl}$ . Take down to complete dryness to render  $\text{SiO}_2$  insoluble, but do not make too hot or  $\text{H}_2\text{SO}_4$  will be driven off. Cool, add 5 c.c.  $\text{HCl}$ , boil off excess, add about 25 c.c. hot  $\text{H}_2\text{O}$ , and boil.

Remove from heat, make alkaline with smallest necessary amount of 1 : 1  $\text{NH}_4\text{OH}$  to precipitate iron and leave supernatant liquid clear. Filter and wash twice with hot water. Put filter paper and contents back into same beaker, break filter with 10. to 20 c.c. hot water, and add just enough  $\text{HCl}$  to dissolve hydrates; put on hot plate a few minutes till solution is effected, then reprecipitate as before with smallest necessary amount of 1 : 1  $\text{NH}_4\text{OH}$ , filter and wash into same beaker containing first filtrate. Make filtrate acid with  $\text{HCl}$ , boil, add a few drops  $\text{HCl}$  (to prevent boiling over when  $\text{BaCl}_2$  is added) and then an excess of boiling  $\text{BaCl}_2$ ; settle, cool, filter on best double filter paper, wash twice with water, once with 1 : 1  $\text{HCl}$ , then 10 times with water; ignite and weigh  $\text{BaSO}_4$ .

### *HCl-soluble Sulfur in Roasted Ore*

Weigh out 1.373 gm. ore, add about 30 c.c. N/10  $\text{HCl}$  solution, boil 10 min., filter, and wash well with hot water. Refilter through a new double paper and wash well. Boil, add a few drops  $\text{HCl}$  and an excess hot  $\text{BaCl}_2$  solution. Settle, cool, filter on double paper, wash, ignite and weigh.

The insoluble sulfur is obtained by difference.

### *$\text{Na}_2\text{CO}_3$ -soluble Sulfur in Roasted Ore*

This is run as a check on the  $\text{HCl}$  method. It gives an insoluble sulfur that is usually about 0.03 per cent. higher than the  $\text{HCl}$ , the notable exceptions being a bad roast on a lime bed, when it may go 0.05 to 0.10 per cent. higher.

Weigh 1.373 gm. ore, boil 10 min. with 30 c.c. N/ $\text{Na}_2\text{CO}_3$  solution, filter and wash well with hot water. Add about 4 c.c.  $\text{HCl}$  to filtrate, or till acid, boil out  $\text{CO}_2$ , make just alkaline with a few drops 1 : 1  $\text{NH}_4\text{OH}$  and boil out  $\text{NH}_3$ . Filter and wash with hot  $\text{H}_2\text{O}$ , acidify filtrate with  $\text{HCl}$ , boil, add a few drops  $\text{HCl}$  and an excess of hot  $\text{BaCl}_2$  solution, settle, cool, filter on double paper, wash, ignite, and weigh.

Class B ores cannot be roasted correctly even approximately by reliance upon visual judgment. The same soluble and insoluble sulfur analyses must be made, but these do not distinguish between the soluble

$\text{CaSO}_4$  and  $\text{CaS}$ , the first of which is harmful principally in setting the sand tanks, while the second interferes greatly with the extraction.

The usual analyses, therefore, combined with our custom of getting only one report a day, proved too slow to prevent bad roasts, and it was found advisable to develop a quick reliable test for soluble sulfides, by means of which a check could be kept on each individual roaster. Arlo C. Greenawalt was detailed to devote his entire time to developing a method. After trial of all available text-book methods, he decided upon the following as the best practical method.

### *Rapid Determination of Soluble Sulfides*

Weigh 15 gm. roasted ore crushed through .80 mesh into a 150-c.c. casserole. Pour onto it 50 c.c. boiling 5-lb. KCN solution, containing 12 lb. NaOH per ton (0.6 per cent.). Boil for 15 min., then filter by aid of a vacuum into a 100-c.c. graduated cylinder. Wash well with hot water and bring volume of filtrate up to 50 c.c. Mix contents thoroughly by inverting the cylinder several times. Extract two samples of 10 c.c. each. To one sample add an excess of  $\text{KAg}(\text{CN})_2$  solution and about 1 gm. dry  $\text{CaO}$ . To the second sample add about 2 gm. dry  $\text{PbCO}_3$ . Heat solutions to boiling, filter and wash well with hot  $\text{H}_2\text{O}$ . Add  $\frac{1}{2}$  c.c. of 1 per cent. KI solution to each filtrate, and titrate with  $\text{AgNO}_3$  (13.038 gm. per liter). Subtract the amount of  $\text{AgNO}_3$  used with the solution to which  $\text{PbCO}_3$  was added, from the amount used with the solution to which  $\text{KAg}(\text{CN})_2$  was added. To obtain percentage of soluble sulfides, multiply this difference by 0.16415.

### *Explanations and Precautions*

The above method is based on the fact that when  $\text{KAg}(\text{CN})_2$  is added to a solution containing alkaline sulfides, a definite amount of free KCN is formed for every atom of silver that combines with the soluble sulfur to form  $\text{Ag}_2\text{S}$ , the equation being  $2\text{KAg}(\text{CN})_2 + \text{K}_2\text{S} = \text{Ag}_2\text{S} + 4 \text{KCN}$ .

It is necessary that the solution of KCN be boiling before adding to the ore, as oxidation of the sulfides will take place in a cold solution. NaOH is added to make the solution alkaline, as results cannot be obtained in acid solution.

A weak KCN solution should be used, as it will take the sulfides into solution readily, and will require a minimum amount of  $\text{AgNO}_3$  for titration.

When  $\text{KAg}(\text{CN})_2$  is added to the solution, the precipitation of  $\text{Ag}_2\text{S}$  is usually incomplete and the precipitate seems to be partly colloidal. The  $\text{CaO}$  is added to coagulate the precipitate, and boiling insures complete precipitation.

The wash water should be hot in order to prevent oxidation of the sulfides to sulfates, which occurs if solutions are allowed to cool.

The addition of KI before titration corrects errors due to the presence of caustic alkalies.

Titration of solution to which  $\text{PbCO}_3$  was added gives the original cyanide present. Titration of solution to which  $\text{KAg}(\text{CN})_2$  was added gives the original cyanide plus the free cyanide that was formed. The difference between the two, or the free cyanide, is directly proportional to the amount of soluble sulfides present in the solution.

The calculation of the value of 1 c.c.  $\text{AgNO}_3$  (13.038 gm. per liter), in terms of percentage of soluble sulfides, is as follows:

$$\text{KCN} = 65.11. \quad \text{KCNS} = 97.18. \quad \text{S} = 32.07.$$

$$1 \text{ c.c. } \text{AgNO}_3 = 0.01 \text{ gm. KCN.}$$

$$65.11 \div 97.18 = 0.01 \div x$$

$$x = 0.01492 \text{ gm. KCNS per c.c. } \text{AgNO}_3.$$

$$32.07 \div 97.18 = x \div 0.01492$$

$$x = 0.004924 \text{ gm. sulfur per c.c. } \text{AgNO}_3.$$

$$\frac{0.004924 \times 100}{15} = 0.03283 = \text{per cent. sulfides.}$$

With 15 gm. ore taken for analysis,  $0.03283 \times 5 = 0.16415 = \text{per cent. soluble sulfides per c.c. of } \text{AgNO}_3$ , as titration took place in 10 c.c. and the total volume was 50 c.c.

If the volume should happen to be larger than 50 c.c., say 57 c.c., the factor is obtained thus:  $5.7 \times 0.03283 = 0.18696 = \text{per cent. soluble sulfides per c.c. } \text{AgNO}_3$ . This method can be run through with nine roaster samples in about  $1\frac{1}{2}$  hr., and, while not absolutely accurate, is the best we have found, and is sufficient for controlling the feeds to the roasters, when the temperatures, as noted above, are maintained.

The limits of insoluble sulfur are: Class A, 0.10 per cent.; class B, 0.15 per cent. The limits of soluble sulfide are: Class A, 0.10 per cent.; class B, 0.15 per cent.

Experience has shown that class B ores give good residues with higher total and insoluble sulfur and soluble sulfide, than class A. The chemical reasons for this are still obscure, but it is possible that the interaction between the sulfates and sulfides in the roasters, as noted above, gives a larger amount of each as a secondary reaction, and these then do not have so deleterious an effect in the treatment.

#### OPERATING CONDITIONS IN THE CYANIDE PLANT

The decision to adopt the above-mentioned more efficient and more delicate treatment necessitated a more carefully planned and flexible cyanide plant, specially arranged to cope with the ore thus roasted. The following are the more important operating factors:

1. Substitution of blanket concentration of gold for plate amalgamation.
2. Elimination of colloids, so far as possible, from the part of the ore to be leached.

3. Ability to change solutions quickly on both sands and slimes.
4. Filling the leaching tanks with a semi-dewatered product.
5. Aeration of slimes soon after classification.
6. More time of agitation.
7. Use of the Crowe vacuum system of precipitation.

1. Amalgamation plates are decidedly more sensitive to unfinished roasting than is cyanide solution. The coarse gold is coated with an alloy which dissolves slowly and is not wetted by quicksilver.

Blankets catch this gold effectively and the particles amalgamate readily when the surface is abraded. The amalgamation treatment includes a small arastra, followed by a grinding pan with universal joint between yoke and muller, and followed by another arastra. The tails are re-blanketed before passing into the main mill flow and the final concentrate is amalgamated in a barrel in strong cyanide solution.

2. The elimination of colloids from the sands is perhaps the most important detail of all. Unless the sands leach exceedingly fast, a "slip-up" in one roaster will cause the complete deoxidation of solution before it reaches the filter bottom, causing a rich residue. If the leaching charge channels perceptibly, the same condition develops in patches. Moreover, in the class B ores there is a marked tendency for the sands to set while leaching. Freedom from colloids distinctly minimizes such tendency.

It was not until 1915 that the more basic ores began to arrive in large quantities and changes in treatment became imperative. There was no available floor space for step classification, and the clumsiness and the expense of a series of cones and pumps were not attractive. The problem was solved by the installation of the Dorr bowl classifier, which makes a separation as efficient, on this class of work, as any ordinary three-stage classification, uses less floor space than was necessary before, requires the minimum of attention, additional solution, and power, and of which the cost for installation and operation are less.

Our plant requirements are: For the slime plant, all material must pass through 100 mesh and not over 12 per cent. remain on 200. For the leaching plant, the sands must be free from colloids.

Between these limits, the feed to the classifiers may vary from the product of two Chilean mills—400 tons of ore—to six Chilean mills—1200 tons—without any classifier adjustments. This is useful, since we control our power peak at the Chilean mills.

3. Change of solution must be made quickly to allow a good margin between an over-roast and an under-roast. On the sands, it is done by the back flow or wash solution in the classifiers.

The slimes flow to a 50-ft., connected-type Dorr tray thickener. The compartments are shallow and the settling capacity always ample. Under these conditions, the slime is quickly passed through as thickened

product. This thickener practically clears itself of slime in half an hour after the feed is shut off. The pulp then goes to a mechanical agitator containing perforated air pipes, where the first change of solution is made.

A further change of solution and partial decantation is made in tray thickeners of the open type. The pulp then passes to agitators.

4. To fill sand tanks by methods such as Butters' distributors, though mechanically cheaper than the present method, has the chemical disadvantage of allowing the sand to rest too long in a dead solution in the early stages of treatment. It might be possible to overcome this, since the sand has been cleaned, by opening the valves and draining the solution off and back to the crushing plant. The possible saving in cost of operation over the present semi-dry method of belt conveyor and distributor is very small, and the method is not as safe from a chemical standpoint.

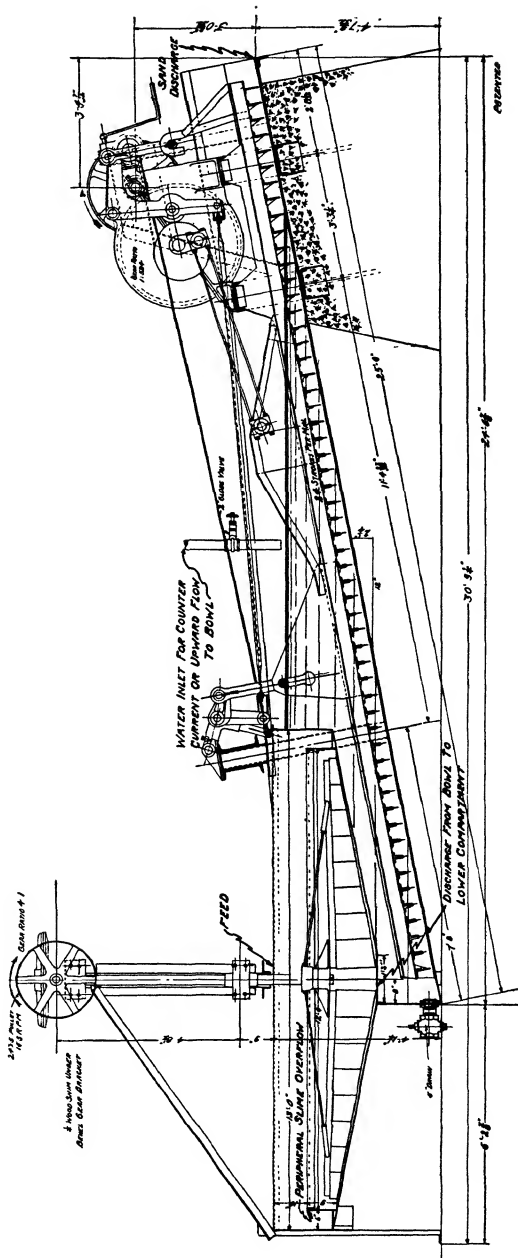


FIG. 1.—ELEVATION OF DORR DUPLEX 8 BY 25-FT. CLASSIFIER EQUIPPED WITH 12-FT. BOWL.

5. Aeration of slimes immediately after classification is very effective—probably such aeration almost instantly gets rid of the unstable soluble sulfides. We attempt to do it fairly fast without additional agitation storage. Possibly, if room permitted, an aerating agitator between classifiers and first settler would be more effective.

6. It is found that a roast giving the lowest residue also gives a slow-treating slime. Therefore excessive agitation is a good safety insurance, and the additional agitation is obtained in a 37 by 23-ft. Dorr agitator, at a cost of 4 mils per ton.

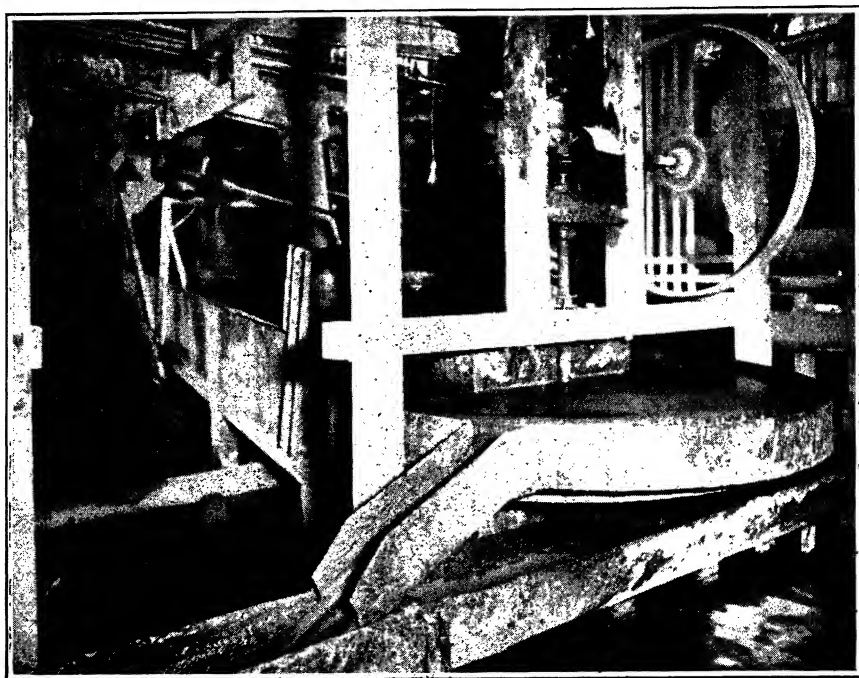


FIG. 2.—DORR DUPLEX CLASSIFIER WITH 6-FT. BOWL.

7. The precipitation of solutions from moderately roasted ores, especially of the more basic type, has been distinctly difficult. A marked improvement in effectiveness and cost has been made by the installation of the Crowe vacuum system of precipitation.

### CONCLUSIONS

There is room for argument regarding the relative efficiency of treating roasted ores in an all-sliming cyanide plant, or in a combination slime and leaching plant. The all-sliming plant is probably less costly to install, and it reduces the number of metallurgical problems. This ore,

however, after roasting, does not require finer grinding than 20 mesh. Further grinding, even in normal times, costs more than the extra recovery attained.

The slimes from a roasted ore have small adhesive qualities and are incapable of sustaining much sand. This limits the type of cyanide apparatus available, unless the all-sliming process is carried far beyond the economical crushing limit.

Undoubtedly, under local conditions, the residues can be dumped far more economically from a combination slime and leaching plant than from an all-slime process.

The ore is high grade and requires a high-strength cyanide solution, thus limiting the effectiveness of the counter-current decantation system, although this system is partially used with good effect in the present slime plant, both as a help in extraction and in washing the values from the filter cakes.

If the sands are sufficiently eliminated from the slime product, the latter is an ideal filtering material; but, even so, the filter department is more expensive to operate, requires more care and more wash solution per ton of ore, and loses more cyanide and gold mechanically than the sand plant.

The deciding feature between the two types of plant lies in the possibility of consistent efficient classification. With poor classification, the advantages lie with the all-sliming process. With good classification, the balance swings decidedly to the combination plant.

The authors were asked to write upon the subject of roasting sulfotelluride ores for cyaniding. The roasting is so interdependent upon the mechanical and chemical details of the cyanide plant that the former is unintelligible without knowledge of the interlocking features of the latter. This is our excuse for the length of detail in this paper.

## DISCUSSION

J. M. TIPPETT,\* Colorado Springs, Colo.—There are several points which offer ground for discussion, although the authors' indicated results leave small chance for argument. Under classification of the ores as they come to the mill, it will be noted that these are classified according to their lime contents. In previous years the classification was based upon sulfur contents, CaO not being considered; class A would be called the oxidized ore and class B the sulfides. Classification according to CaO is the first public indication that a decided change in the ore has necessitated improved methods of treatment. The very serious effect that CaO, even in small quantities, has upon extraction has been noted for some years.

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\* Superintendent, Colorado Springs Milling Dept., Portland Gold Mining Co.



The bedding of the ore is an interesting point; 66 per cent. of the ore, containing not more than 2 per cent. CaO, is called Bed A, while 34 per cent., containing over 2 per cent. CaO, goes to make Bed B. Why could not all the ore be put into one bed in which the CaO would average approximately  $2\frac{1}{2}$  per cent.? This would obviate the necessity for regulation, and would avoid poor roasts and loss of tonnage. No doubt there are good reasons why this is not done; first, can the two classes of ore be uniformly mixed? If this could be accomplished, then feed and heat conditions in the roaster could undoubtedly be regulated satisfactorily. Second, if the class A ores contained more than average sulfur, the ill effect of mixing it with a high CaO ore would be more pronounced.

Since roasting is the most important step in the treatment of these ores, it may pay to examine the different types of mechanical roasters in general use. These are the Holthoff straight-line, the Pierce turrett, and the Edwards. In the first two roasters, the rabblers carry the ore forward through the roasting hearth in practically the same manner. The Edwards roaster operates quite differently, in that the ore, on its passage through the roasting hearth, passes from one side of the roaster to the other many times before it is discharged, thereby increasing the time during which the ore is subjected to heat.

In describing the process of rabbling, it is stated that with 27 pairs of rabblers, making 3 r.p.m., the time the ore is passing through the roaster is 6 hr. In the Pierce and Holthoff roasters the maximum time is 3 hr. The greater length of time during which the ore is subjected to heat, and the rapid rabbling which exposes fresh surfaces of ore more frequently, are two important features of the Edwards roaster which the other types do not possess.

The remarkably poor sand residues are an indication of good roasting followed by careful classification. It would have been interesting to state the assay of the residues from the product finer than 100-mesh, which is approximately 30 to 50 per cent. of the total ore treated. Part of this product consists of what I would term the natural slime in the ore, existing as talc and vein matter, which generally contains values. It is this product, together with the portion finer than 100-mesh made in crushing before roasting, upon which it is difficult always to obtain a low-grade residue.

A. L. BLOMFIELD (author's reply to discussion\*).—Mr Tippet's questions are to the point. The classification of the raw ore above and below 2 per cent. CaO has been arrived at by practical results. We have found that the high-lime beds are not improved by mixing with the so-called siliceous ores, while the residues from the latter come up to the high-lime level when even a small proportion of high lime is present.

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\* Received Apr. 28, 1919.

Time and again the plant has been run with beds containing all the ore mixed, but the results have never been as good as when the high-lime ores are run separately. Since the paper was written, the ores forming beds of over 5.5 per cent. CaO have increased from 34 per cent. to 55 per cent. The sulfur content is practically the same in each type of bed.

Mr. Tippet's comparison of the Holthoff and Pierce types of roaster with the Edwards, bears out our opinion. The former have longer periods between rabbling with more chance of overheating and sintering the exposed surfaces.

The value of the slime-plant residues on meshes finer than 100 corresponds closely to those of similar mesh in the sand tanks given, except that the minus 200-mesh product is between 10 and 20 c. higher. This is due to this minus 200-mesh material being largely colloidal whereas the minus 200 in the leaching plant is a clearly defined sand; it is invariably the rule on Cripple Creek ores that the colloid residues contain the most insoluble gold.

Manufacture of Silica Brick\*

BY H. LE CHATELIER AND B. BOGITCH

(Milwaukee Meeting, October, 1918)

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INTRODUCTION

SILICA brick are indispensable in the manufacture of steel because they alone are able to withstand the high temperature of regenerative furnaces. All attempts to replace silica brick by other refractory materials for this purpose have failed, but the reason for this failure has remained obscure; we shall give an explanation later in this paper.

Before the war, silica brick employed in France came principally from abroad, for which reason, since the beginning of hostilities, certain French steel works have been seriously handicapped. Our attention was first directed to the question by M. Bied, Engineer of the Teil Works. With him, we began certain investigations, at first using our laboratory and the furnaces of neighboring steel works. The larger part of our experiments, however, have been made in the laboratory of the Faculty of Sciences at the Sorbonne. For the heating of our samples, we are deeply obliged to MM. Charpy, Yeatmann and Guérineau. In undertaking these studies, our first aim has been to render assistance to French industry, by indicating, as precisely as possible, the necessary conditions for the manufacture of high-grade brick; but at the same time we have

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\* This paper was submitted to us in manuscript form by M. Le Chatelier, who called our attention to the fact that it had already been published in the *Bulletin de la Société de l'Industrie Minérale*, 3d livraison de 1917, page 49. On account of this previous publication, it has not seemed necessary to reproduce the paper in our *Transactions* in the form of a literal translation, and the editor has therefore not attempted to conform exactly to the language of M. Le Chatelier. The importance of silica brick in the American steel industry, and the eminence of M. Le Chatelier in this field, together give ample justification for the reproduction of this paper in the English language.

intended to give an example of the manner in which scientific methods may be put to practical application in the solution of industrial problems. Although our work has been confined to samples of only a few grams, we are nevertheless convinced that our information can be directly utilized by brick works of which the output is measured in thousands of tons.

### METHODS OF INVESTIGATION

As to what are the most important and necessary qualities of silica brick, if one were to ask the steel metallurgists they would almost unanimously reply that there was only one, namely, that the brick should permit the greatest possible number of runs without necessity for repairing the furnace roofs; they might suggest 400 runs as a satisfactory figure. Durability, however, is not the only factor. The brick have to be transported from the factory to the steel works without being injured by the jar or by freezing; many otherwise satisfactory bricks will not satisfy this last condition. It is further necessary that the brick shall not be too expensive, in order not to add unnecessarily to the price of the manufactured steel. The fundamental requirements, therefore, are the following: (1) durability of the furnace; (2) durability in transport; (3) moderate price.

### *Destruction of Furnace Roofs*

Considering first the subject of durability of furnaces, the processes by which furnace roofs are destroyed can be answered by the direct observation of those in charge of furnaces. Our inquiries along this line did not meet with great success; only two steel works, the Ruelle Foundry, and the Chaussade Works, have been able to give us precise information, but unfortunately contradictory. One of these works has assured us that the roofs of Martin furnaces are almost invariably destroyed by progressive decrepitation under the action of heat. The other works, on the contrary, has assured us that similar furnaces are destroyed almost exclusively by melting. The brick manufacturers, on the other hand, have given us still a third reason, the ignorance or negligence of those in charge of the construction and operation of the furnaces. The engineer may have designed the profile of the furnace badly, not placing his burners in the most desirable place, or applying the first heat of the furnace too abruptly. The builder may have erred in shaping his bricks carelessly and laying them irregularly, so as to produce local pressure sufficient to occasion rupture. Above all, the heat may melt the roof of the best furnace, sometimes in a few hours, if the gas is badly regulated or if the reversals of flame are at too long intervals. From the discussion of this contradictory information and from suggestions found in different

foreign publications we have arrived at the provisional conclusion that the destruction of the roofs of steel furnaces may arise from five possible different causes, ranking in the following order of diminishing importance:

1. Superficial spalling of the brick under the influence of the highest temperatures developed in the furnace. This phenomenon is often noticed in the form of a continuous rain of little fragments, the falling of which may, after a few days, lead to the entire disappearance of the brick. This source of destruction depends upon two properties of the brick: Expansion of silica under high temperatures, and lack of mechanical strength at high temperature.

2. Superficial melting of the brick. Brick always melts superficially and continuously under the action of the spattering slag; this normal destruction of brick may continue for several months before leading to an actual cavity in the roof. Often, however, the brick will melt all at once for a considerable width, several centimeters at a time, by which means the normal destruction of the brick may be multiplied by 10 or more. This phenomenon depends upon the fusibility of the brick itself and upon its permeability, which facilitates the absorption of slag.

3. Flaking or shelling of the brick in the less intensely heated region. This begins to occur during the warming of the masonry, and below red temperature, and continues in the more remote parts after the furnace as a whole is considerably hotter. The brick, thus fractured, may then become detached and fall from the roof. This phenomenon is caused by the excessive dilation which is shown by all crystalline silicas at their temperatures of reversible transformation. Quartzose rocks decrepitate at a temperature of about  $570^{\circ}$ ; cristobalite, around  $230^{\circ}$ , undergoes an abrupt change in dimensions, of very important character; tridymite, finally, at about  $150^{\circ}$  undergoes a change of slight importance. This tendency to rupture is offset by mechanical resistance of the brick and by its structure, that is to say, by the size of its grain and its amount of porosity.

4. Dislocation of the roof by excessive expansion. In furnaces made of silica brick, the roof always rises more or less when the furnace is first put into operation; this rising often becomes excessive and very irregular from point to point, which then leads to the falling in of the roof. This dislocation results, the same as spalling, from expansion of silica. If the brick is sufficiently resistant, and is heated over a considerable width all at once, it does not spall but causes the roof to rise.

5. Collapse of the roof. The frequency of this accident with clay, magnesia, and alumina brick, makes it impossible to use these materials for the construction of the roofs of furnaces intended to maintain very high temperatures. Collapse will also occur, but very rarely, in furnaces made of silica brick. Collapsing results from the softening that precedes fusion and therefore depends upon the same factors as fusibility. It is

very rare in silica brick, being counterbalanced by the expansion of the quartz resulting from its transformation into silica of low density.

The reasons for the destruction of furnace roofs, and the properties of the brick upon which these depend, can now be tabulated as follows:

CAUSES OF DESTRUCTION	PROPERTIES OF BRICK
1. Spalling	1. Fusibility
2. Fusion	2. Compressive strength at high temperatures
3. Flaking or shelling	3. Permeability
4. Dislocation of the roof	4. Expansion
5. Collapse	5. Dilation
	6. Compressive strength at ordinary temperatures.

It would be hazardous to assert that this list is absolutely complete, but if any phenomenon has been omitted it is for the engineers of steel works to inform us.

### 3. THE TRIDYMITITE NETWORK

It is well known that silica exists under five different allotropic forms, as shown in Table 1.

TABLE 1.—*Allotropic Forms of Silica*

	Density	Temp. of Transformation, °C.	Abrupt Change in Linear Dimensions, Per Cent.
Quartz.....	2.65	570	0.25
Cristobalite.....	2.34	225	1.00
Tridymite.....	2.27	150	0.10
Chalcedony.....	2.58	570	0.10
Glass.....	2.22		

Quartz is the universal raw material for the manufacture of silica brick. Deposits of quartz can be grouped into four distinct classes: (1) Quartz veins, consisting of large crystals adjoining one another, forming translucent or opalescent white masses. (2) Quartzite (Figs. 3, 4, 5, and 6), metamorphic rock in which the grains of quartz are so strongly cemented together that upon breaking the rock the fractures traverse the grains of quartz rather than their boundaries. Quartzite contains impurities in variable proportion, sometimes lodged between the grains, like mica, sometimes included in the quartz crystals themselves, as oxide of iron, for example. (3) Sandstone (Figs. 1 and 2), the grains of which are combined by a cement having but little resistance, in which fracture

occurs by the separation of the grains, giving a dull luster to the fractured surface. (4) Sand, the grains of which are separated. The purity of sand



FIG. 1.



FIG. 2.

FIG. 1.—CARBONACEOUS SANDSTONE FROM SHEFFIELD; GANISTER, USED FOR THE MANUFACTURE OF SILICA BRICK. DISSEMINATION OF MICA BETWEEN THE QUARTZ GRAINS. NATURAL LIGHT; MAGNIFICATION,  $\times 136$ .

FIG. 2.—SAME SAMPLE AS FIG. 1, UNDER POLARIZED LIGHT.

is very variable; that of Fontainebleau analyses 99.5 per cent. silica; after this come the yellow argillaceous sands, and finally sandy clays.



FIG. 3.



FIG. 4.

FIG. 3.—SOUVIGNY QUARTZITE, WITH DULL FRACTURE. MATERIAL OF FIRST QUALITY. POLARIZED LIGHT; MAGNIFICATION,  $\times 136$ .

FIG. 4.—SOUVIGNY QUARTZITE, WITH RIBBON STRUCTURE. MATERIAL OF SECOND QUALITY. CHALCEDONY CONCRETIONS ABOUT THE QUARTZ GRAINS. POLARIZED LIGHT; MAGNIFICATION,  $\times 136$ .

An important character of sand is the uniformity in size and shape of its grains, which are objectionable features for the manufacture of brick.

Most factories employ quartzite containing not more than 3 per cent. of basic oxides, and mix with it 2 per cent. of lime. The crushing of the rock is conducted in such manner as to preserve a number of large grains, having a maximum size of between 5 and 10 mm. The firing is performed at a high temperature, much above that employed in the manufacture of clay brick, and is maintained for a much longer period. This temperature may vary from  $1350^{\circ}$  to  $1450^{\circ}$  according to the nature of the quartz and the ease of its transformation.

Firing progressively reduces the density of the silica; quartz transforms first into cristobalite and finally into tridymite, as one of us has shown 25 years ago. These are the basic facts upon which our researches were conducted.



FIG. 5.

FIG. 5.—QUARTZITE CONTAINING CHALCEDONY. THIS DISINTEGRATES ON FIRING AND CANNOT BE USED FOR THE MANUFACTURE OF SILICA BRICK. POLARIZED LIGHT; MAGNIFICATION,  $\times 136$ .



FIG. 6.

FIG. 6.—QUARTZITE WITH DEFORMED GRAINS, CEMENTED BY OPAL OR CHALCEDONY. UNDESIRABLE FOR THE MANUFACTURE OF SILICA BRICK. POLARIZED LIGHT; MAGNIFICATION,  $\times 136$ .

### *Compressive Strength of Refractory Products*

Before attacking the problem of the manufacture of silica brick, we have sought to answer an allied problem: Is it possible to find certain measurable properties of refractory products which will explain the superiority of silica over clay, alumina, and magnesia? An exact knowledge of the reason for the superiority of silica brick would certainly be a valuable guide to determine what properties are the most important to develop in the manufacture of refractory materials. One difficulty in explaining the superiority of silica brick has been that in previous experiments it has been noted that the melting point of silica brick was not higher than that of other refractory products. Kaolinite melts at



1800°, the same as silica; alumina and magnesia melt at much higher temperatures. These latter materials should, therefore, afford brick at least as good and perhaps better than quartz. Instead of determining simply the melting point, as has often been done before, that is to say, the temperature at which the material yields under a pressure of only a few grams per centimeter, we decided to measure the compressive resistance of these materials throughout the whole field of temperature.

The experiments were made in a small furnace, heated by a blast of illuminating gas. The furnace, having an interior capacity of 500 c.c., gave a temperature of 1600° at the end of half an hour, with a gas consumption of 3 cu.m. per hour. The walls of the furnace were made of corindite (melted bauxite) cemented with a little sodium silicate; this material seemed to resist the action of heat indefinitely provided it was not required to support any load. The samples to be tested were in the form of little cubes, 1 cm. on a side, cut out of the bricks to be studied. Pressure was transmitted to them through a bauxite cylinder previously heated to a temperature of 1600°, penetrating the roof of the furnace. The temperature was measured by a thermo-electric couple attached to the sample. The results were as shown in Table 2.

TABLE 2.—*Crushing Strength of Bricks at Different Temperatures*  
(Pressures in kg. per sq. cm.; temperatures in ° C.)

	15°	500°	1000°	1300°	1400°	1500°	1600°
Silica (Star brand).....	170	150	120	75	60	48	30
Kaolin.....	190	180	210	90	(12)	(1)	(0.5)
Eubœan magnesia.....	420	380	320	270	240	(185)	(8)
Styrian magnesia.....	145	130	85	66	(5)	(3)	(1)

It is thus apparent that at 1600°, which is still 100° below the temperature of the Siemens-Martin furnace, silica brick has a compressive strength very much higher than that of the other refractory products. Furthermore, a factor which is of no less importance, the silica brick broke abruptly at all temperatures; they did not register any progressive deformation before rupture. With clay and magnesia brick, on the other hand, the observations were entirely different. At temperatures below 1000° they broke abruptly like rigid bodies, but at higher temperatures, above a limit varying according to the purity of the material, the brick yielded little by little, like plastic matter. If, instead of making these tests in about one minute's time, as in our investigations, the application of the force had been prolonged for 1,000,000 times longer, as occurs in the roofs of furnaces, the brick would have yielded under pressures so small as to be practically negligible, having somewhat the character

of rosin at ordinary temperature. In order to rupture such materials abruptly, an impact is necessary; left to themselves, they yield under their own weight and spread out level like a liquid.

The transformation from complete solidity to complete viscosity is obviously progressive. Within a certain range of temperature, an initial

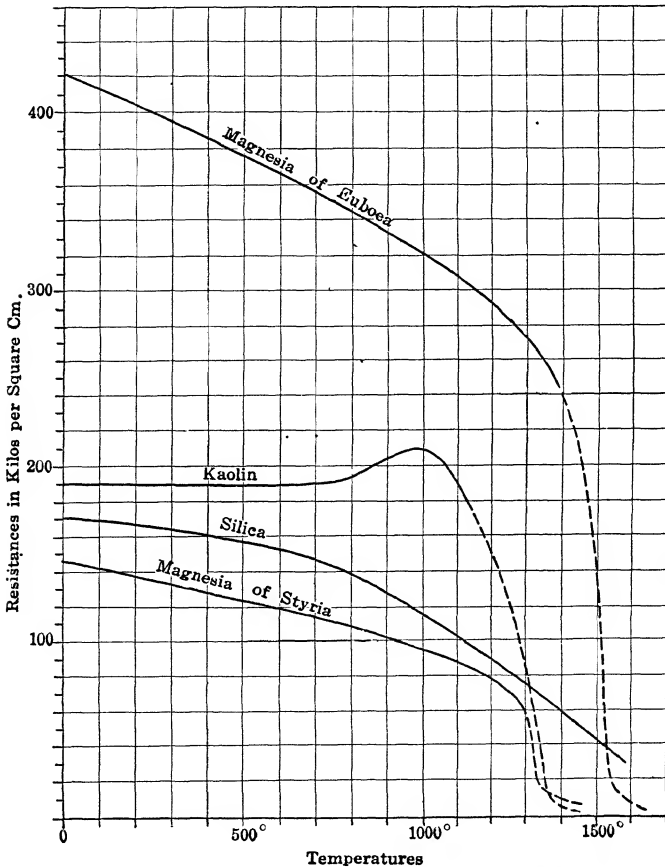


PLATE I.—VARIATION IN THE CRUSHING STRENGTH OF REFRACTORY PRODUCTS AT DIFFERENT TEMPERATURES.

deformation is followed by a true rupture. Complete viscosity is definitely attained above the following temperatures:

	DEGREES C.
Silica brick.....	1700
Euboean magnesia .....	1500
Styrian magnesia .....	1300
Kaolin brick.....	1300
Ordinary refractory brick.....	1200

It is very easy, in the test, to distinguish between these two methods of rupture. In the case of complete solidity, the sample breaks abruptly and the whole compression apparatus comes together with a jar. In the case of viscosity, the beam of the pressure machine falls progressively and continuously. The sample is first compressed upon itself, by the closing of its spaces, then it expands in the form of a barrel, then bursts on its periphery, and finishes by breaking or not, according to its solidity. In the intermediate stage between solidity and complete viscosity, the two methods of deformation can be successively observed. The sample first yields progressively, then breaks more or less abruptly, throwing out fragments. The remaining mass has no further solidity; on the contrary, the fragments thrown out by the bursting of completely viscous material resume their original solidity after cooling.

### *Recrystallization of Silica*

This ability of silica brick to preserve their rigidity at the very highest temperatures explains their superiority for furnace manufacture. As a means of reducing this factor to its simplest terms, we began with an examination, under polarized light, of thin sections cut from bricks of good quality, one marked "American Star" and the other marked "Imphy," these being the two bricks which showed the highest compressive resistance at 1600°, namely 30 and 40 kg. per sq. cm. They were composed of large, easily recognizable grains of the original siliceous rock, completely transformed into cristobalite and surrounded by a magma formed of little elongated crystals of tridymite (Fig. 7). The rigidity of the brick is evidently due to the crystallization of tridymite, which forms a continuous network, in the meshes of which the fusible silicates are lodged. The presence of these latter materials does not detract from the solidity of the mass any more than water in the cells of pumice stone would diminish its strength; in both cases the solid network is unbroken.

In magnesia brick, on the contrary, and also in clay brick, at least in those manufactured under ordinary conditions, this recrystallization does not occur. The slightly fusible material, magnesia, forms isolated grains which are surrounded, at ordinary temperatures, by a magma of very solid silicates; the latter, however, melt at around 1300°, if ferruginous, or at about 1500° if purely magnesian. Above these temperatures, therefore, the solid grains swim in a melted mass and can slide on one another; the more fluid the magma, the more readily they slide.

The formation of this continuous network of silica is exactly parallel to the phenomenon observed in the hardening of cement. A mixture originally consisting of isolated grains, when tempered with water, is progressively transformed by chemical reaction into a coherent mass.

In the same manner, the isolated grains of quartz in a brick become set on contact with melted silicates, which operate as solvent.

Quartz is unstable at temperatures above  $800^{\circ}$ , but owing to its remarkable passive resistance, it is able to remain for a long time in that condition at very high temperatures, even up to  $1600^{\circ}$ . If it is then brought into contact with a solvent, melted silicates for example, it dissolves in that with a readiness very much greater than that of the more stable forms of silica, cristobalite and tridymite. This is due to the unanswerable and thoroughly established laws of physical chemistry. The quartz thus gives rise to supersaturated solution, from which one of the more stable varieties soon begins to crystallize. The melted mass, now being



FIG. 7.

FIG. 7.—WELL FIRED AMERICAN BRICK, WITH NETWORK OF TRIDYMITÉ BETWEEN THE QUARTZ GRAINS, WHICH ARE ALMOST COMPLETELY TRANSFORMED INTO CRISTOBALITE. POLARIZED LIGHT; MAGNIFICATION,  $\times 136$ .



FIG. 8.

FIG. 8.—ASSAILLY BRICK, REMAINING FOR A YEAR IN ONE OF THE FLUES OF A MARTIN FURNACE. COMPLETE TRANSFORMATION INTO COARSE-GRAINED TRIDYMITÉ, WITH CHARACTERISTIC INCLUSIONS. POLARIZED LIGHT; MAGNIFICATION,  $\times 136$ .

no longer saturated with respect to quartz, is able to dissolve additional quantities of it. Gradually, therefore, the entire amount of quartz recrystallizes into the variety that is most stable at high temperatures, tridymite. In practice, if the firing of silica brick has not been sufficiently long, the proportion of cristobalite, and sometimes even of quartz, is greater than that of tridymite in the finished product, if of poor grade. Burning for almost a month at the highest temperature of the steel furnace is necessary to transform silica completely into tridymite. The crystals of tridymite thus formed by solution attach themselves to one another, as is always the case under similar conditions, and form the network above mentioned.

*Quality of Brick*

This explanation for the superiority of silica brick gave rise to the question whether variations in quality were not exactly parallel to the compressive strength at high temperatures. To settle this question, we requested, from different steel works, samples of brick which had been used for the construction of furnaces, as to the relative quality of which they were able to advise us. The Ruelle and the Guérigny Works sent us well classified series of samples upon which we made tests giving the results shown in Table 3. The tests on compressive strength at high temperatures were made, with some samples as soon as  $1600^{\circ}$  had been



FIG. 9.



FIG. 10.

FIG. 9.—TRIDYMITE CRYSTALS SURROUNDED BY A SOLUTION OF MAGMA. BRICK OBTAINED ON DISMANTLING THE ROOF OF A MARTIN FURNACE. COLUMNAR CRYSTALS AND RECTILINEAR CLEAVAGES CHARACTERISTIC OF TRIDYMITE. NATURAL LIGHT; MAGNIFICATION,  $\times 136$ .

FIG. 10.—CRISTOBALITE CRYSTALS SUBMERGED IN A TRANSPARENT GLASS. MATERIAL DERIVED FROM RELINING OF A BESSEMER CONVERTER AT SHEFFIELD. CIRCULAR CLEAVAGES CHARACTERISTIC OF CRISTOBALITE. NATURAL LIGHT; MAGNIFICATION,  $\times 136$ .

reached, while with other samples only after they had been maintained at this temperature for one hour. We realized that these two methods of proceeding might give different results in some cases. At the same time, we made determinations of a number of other physical properties, such as absolute density, apparent density, weight of sulfate corresponding to basic oxides present, and finally compressive resistance at ordinary temperatures.

The first brick, Assailly, had remained a year in the lining of the outlet from a gas producer. The next two bricks, American Star, and G. I., of French manufacture, were recommended to us as of particularly good quality. The last brick in the Table, RSG, it has not been possible to use,

a roof constructed with these bricks having melted on the first application of heat. The other bricks were of French, English, and German manufacture.

TABLE 3.—*Features of Certain Silica Bricks*

Mark	Quality	Sulfates, Per Cent.	Density		Crushing Strength		
			Absolute	Apparent	Temp., ° C.	Time, Min.	Kg. per Sq. Cm.
Assailly.....	Very good	13.6	2.30	1.92	15	..	550
Assailly.....	Very good	....	....	..	1600	60	90
Star.....	Very good	9 06	2 33	1.66	15	..	170
Star.....	..	....	....	....	1600	5	33
Star.....	..	....	....	....	1600	60	30
G.I.....	Very good	8 40	2.33	1 88	15	..	185
G.I.....	..	....	....	....	1600	60	41
R.B.....	Very good	13 10	2 35	1.60	15	..	62
R.B.....	..	....	....	....	1600	60	9.5
R.L.....	Very good	14 3	2 40	1.85	15	..	265
R.L.....	..	..	....	....	1600	5	41
R.L.....	..	....	....	....	1600	60	25
G.A.....	Good	14 0	2 40	1.77	15	..	190
G.A.....	..	....	....	....	1600	60	21
D.....	Good	8 4	2.45	1.73	15	..	320
D.....	..	....	....	....	1600	5	55
D.....	..	....	....	....	1600	60	20
G.A.1.....	Very bad	14 5	2 46	1 80	15	..	252
G.A.1.....	..	....	....	....	1600	60	4.4
R.F.....	Very good	13 7	2 48	1.84	15	..	195
R.F.....	..	....	....	....	1600	..	11
G.A.2.....	Very bad	12 8	2.48	1.78	15	..	148
G.A.2.....	..	....	....	....	1600	60	5
G.M.....	Medium	9.5	2.53	1.84	15	..	84
G.M.....	..	....	....	....	1600	5	17
G.M.....	..	....	....	....	1600	60	2
R.L.....	Bad	9.75	2.56	1.94	15	..	350
R.L.....	Shatters	....	....	....	1600	5	18
R.L.....	..	....	....	....	1600	60	4.5
R.S.G.....	Bad	25.0	2 56	1.73	15	..	57
R.S.G.....	(melted)	....	....	....	1550	60	22
R.S.G.....	..	....	....	....	1660	Melted	

All of the good bricks, after being held at 1600° for one hour, showed a compressive strength greater than 10 kg. per sq. cm.; most of the good bricks exceeded 20 kg., while the poor bricks were below 5 kg. It seems evident, therefore, that rigidity at high temperatures is the most essential if not the only important quality of silica brick. Most of the good bricks have densities below 2.40, the very good ones being as low as 2.33. The amount of sulfate in good bricks is below 15 per cent.

## INDEPENDENT VARIABLES

Table 4 enumerates the essential properties of silica brick, together with the elementary factors to which they are more or less directly related:

TABLE 4

PROPERTIES OF BRICK	ELEMENTARY FACTORS
1. Fusibility	1. Nature of quartz
2. Compressive strength at high temperature	2. Size of particles
3. Permeability	3. Nature of fluxes
4. Expansion	4. Proportion of fluxes
5. Dilation	5. Thoroughness of mixing
6. Compressive strength at ordinary temperature	6. Tempering water
	7. Pressure of molding
	8. Temperature of heating
	9. Duration of heating

*Fusibility*

Fusibility depends, in the first place, upon the presence of basic oxides mixed with the quartz. Secondly, which may appear somewhat paradoxical, fusibility depends upon the conditions of manufacture; certain foreign manufacturers go so far as to assert that fusibility depends much more on the texture of the brick and their manner of firing than on their chemical composition.

The quartz employed for silica brick is almost never pure, generally containing mica; furthermore, the crushing of the quartz always introduces a certain proportion of iron. The average composition of silica, crushed and ready for use, is as follows: Alumina, 1.5; oxide of iron, 1.0; magnesia and alkalies, 0.5; silica, 97 per cent. As a binder, lime to the extent of 2 per cent. is always added, thus making a total of 5 per cent. of basic oxides. If this is computed to the condition of sulfate, after attack by hydrofluoric acid, it represents a total of 12 per cent. of sulfate; bricks containing above 15 per cent. of sulfate are useless for steel furnaces, and, in general, good bricks should not contain more than the equivalent of 10 per cent. sulfate.

The influence of the different basic oxides on increase of fusibility should not be exactly alike, but on this point our studies have not been conclusive. Oxide of iron seems to have the least influence, since silicates of iron are decomposed at high temperatures; alumina comes next, then lime, and finally alkali, the action of which, even in a small proportion, appears to be very energetic.

The effect of the method of manufacture upon the fusibility of the brick is unquestionable. Bricks having a perfectly normal chemical composition are often found to fuse at the temperature of steel furnaces,

giving rise to a granular mass in which the large grains of silica are mobile. This arises from the absence of a sufficient amount of fine material in the mixture, and from a firing not sufficiently complete to permit the development of a proper network of tridymite. On further heating, the network disappears by solution in the magma, leaving between the large grains a glass which is less siliceous and more fusible in proportion to the absence of fine grains. The examination of thin sections of brick shows very clearly that the grains of silica are attacked by the magma only to a depth of 0.01 to 0.02 mm. Only grains of 0.03 mm. or less dissolve completely in the magma and contribute to the formation of the network. Assuming that the amount of grains of this size is only 10 per cent. of the total and that 2 per cent. of lime is added; this would represent 20 per cent. of the weight of the fine grains and would give a fusible glass in which the large grains would swim. According to our investigations, the proportion of impalpable material, that is to say, of quartz passing through a screen of 4900 openings per sq. cm. (approximately 200-mesh), should be at least 25 per cent. Adopting that proportion, at the very first test we obtained a compressive strength of 30 kg. per sq. cm. after heating for one hour at 1600°, which is comparable to the strength of the best commercial brick.

### *Shelling or Rupturing*

Shelling or rupturing of brick at low temperatures, which occurs on the external surface of brick at the beginning of heating, but may penetrate half way through the brick toward the end of the heating, arises principally from the change in volume which the different varieties of silica undergo during their reversible transformation. The transformation of cristobalite, accompanied by a linear expansion of 1 per cent., is by far the most serious. Every time a brick containing a large proportion of cristobalite passes quickly through the temperature of 225°, crevices are produced, which diminish the rigidity of the brick. According to the experiments of J. Spotts MacDowell,<sup>1</sup> a single heating to above this temperature, followed by cooling in the air, reduces the compressive strength of silica brick by 50 per cent. For this reason, it is impossible to utilize silica brick in furnaces which will be allowed to cool periodically; under such conditions, the brick will become fissured and almost completely disintegrated in a short time. In steel furnaces maintained under constant heat, this accident is most likely to occur during the period of preliminary heating.

This disadvantage of silica brick is offset by their mechanical strength, opposing the production of crevices, by the presence of large grains of

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<sup>1</sup> *Trans.* (1917) 57, 3-59 (contains bibliography).



quartz, the large spaces between which prevent the spreading of the crevices, but, above all, by as complete a transformation as possible of cristobalite into tridymite.

A very striking experiment, which can be strongly recommended to all steel-works engineers, consists in placing a silica brick in a small gas furnace, or a kitchen stove, and regulating the heat in such a manner that the lower surface of the brick is raised to an incipient red in 5 min.; after maintaining the heat for a quarter of an hour, the brick is taken from the fire and allowed to cool for the same length of time. After this treatment, fragments can be broken off from the brick by hand, and the same operation can be repeated. In the case of a brick having absolute density of about 2.35, the brick will have been entirely disintegrated at the end of the fifth operation.

On the other hand, a slow and regular heating of the brick prevents this disintegration by diminishing the temperature gradient toward the interior of the brick. We have been able to avoid all fissuring with a brick which fractured easily, under the above treatment, by heating it to 500° at the rate of 50° per hour, and allowing it to cool at the same rate.

Wood fires, which are frequently employed for starting the operation of a steel furnace, are particularly dangerous by reason of the irregularity of their heating effect, regions of high temperature necessarily occurring above the points at which combustion is most active. It would be desirable to avoid wood fires at steel works, as has been done at glass works, by using currents of hot air with progressively increasing temperatures.

### *Expansion*

Expansion, leading to the superficial spalling of brick and warping of roofs at high temperatures, is a function of the following factors:

1. Change in the condition of silica from that of quartz to a material of less density.
2. Mechanical strength of the brick, opposing expansion.
3. Porosity of the brick, permitting expansion to accommodate itself in the spaces between grains.
4. Rate of increase of temperature during heating. Abrupt heating may produce expansion three to six times as great as a slow heating, such as is maintained in well conducted kilns.

When the change from quartz to tridymite occurs in a massive unfractured block, it causes a linear expansion of about 5.5 per cent. However, by a sufficiently slow firing, the apparent expansion can be reduced to 2 per cent., that is to say, a value below that of absolute expansion. This is accompanied by a parallel diminution in the volume of voids. On the other hand, if the heating during the first firing is too abrupt, and still more if, after an insufficient firing, the transformation is concluded

only in the steel furnace, the linear expansion may reach 10 per cent.; there would then be a considerable increase of porosity, rather than a reduction. The brick would become both less strong physically and also more permeable to slag, to say nothing of the ruptures produced directly by the expansion.

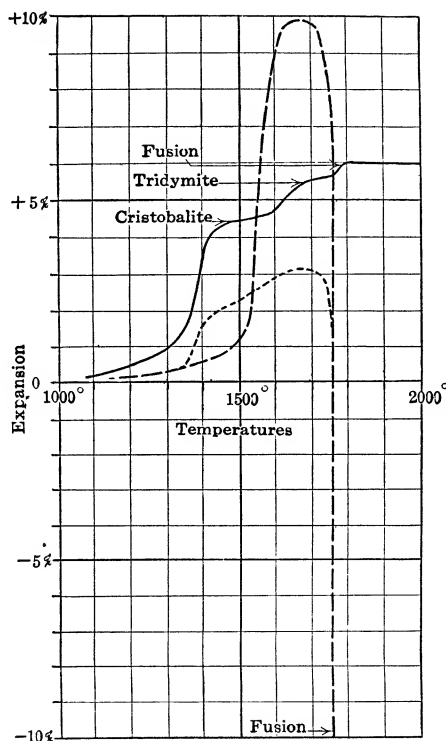


PLATE II.—EXPANSION OF SILICA BRICK UNDER VARYING CONDITIONS.

In the accompanying diagram (Plate II), the solid line represents the theoretical linear expansion of a compact mass. The dotted line shows the normal expansion of a well fired brick not introduced into the steel furnace until after the transformation of the quartz; in this case, one should expect a minimum expansion of 2 per cent. in the kiln, and of 1 per cent. in the steel furnace. The broken lines represent the expansion of a badly fired brick inserted prematurely and abruptly into a steel furnace.

#### *Transformation of Quartz*

The expansion of brick, which is intimately related to the transformations of quartz, is of the greatest importance from the point of view of the manufacture of silica brick, and of their use in steel furnaces. Our experiments on this subject have not offered so complete a solution of the problem as we could have desired.

Day's experiments<sup>2</sup> have shown that quartz ceases to be stable above 800°. Its enormous passive resistance, however, opposes this transformation and retards it to such an extent that after heating to 1600°, for only a short time it is true, fragments of unaltered quartz can often be found. In order to produce transformation below 800°, it is necessary to apply some special solvent, such as vanadate of sodium, and to prolong the action of the heat for several days.

In the manufacture of silica brick, the transformation of quartz occurs in three different ways:

1. By solution and recrystallization in the melted silicates. This phenomenon begins at the melting point of these compounds, around 1200°, and proceeds with rapidity in proportion to the temperature. It is this reaction that produces the network of tridymite referred to above.

2. By direct transformation of quartz fragments without any other agencies than temperature and the presence of natural impurities in the rock. This transformation takes place at variable temperatures in quartz from different localities; the exact reason for the variation is not understood. Silex can be transformed in less than an hour at 1300°; the Fontainebleau sand, which is very pure, transforms at 1500°; the impure quartzite ordinarily used for the manufacture of brick transforms at temperatures between these two limits.

3. By direct transformation of the quartz under the influence of foreign bodies which seem to penetrate by diffusion to a certain depth into the grain. Cristobalite, according to some investigators, would be particularly likely to give rise to these solid solutions.

One of us has shown, 25 years ago, that crystals of very pure quartz can be put through a furnace for the production of hard porcelain, without undergoing any transformation. Under the same conditions, the same quartz finely pulverized is completely transformed into silica of low density. In the same manner, in the manufacture of fine faience, the more finely the quartz is pulverized, the more complete is its transformation. This influence of grain size cannot be explained except on the assumption of a superficial action from vapors in the atmosphere of the furnace, whether steam, or alkali vapors derived from other elements in the ceramic mixture, notably feldspar.

We have made several tests on the transformation conditions of quartz, and have studied successively the influence of the following factors:

1. Nature of the quartz.
2. Size of the fragments.
3. Nature of the flux.
4. Proportion of flux.
5. Length of firing.
6. Temperature of firing.

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<sup>2</sup> *American Journal of Science*, ser. 4 (1914) 37, 1-39.

The tests were made in a Bigot furnace, the several samples being heated simultaneously. Except for the last experiment, regarding the influence of temperature, the heating was always conducted in such manner as to raise the temperature to  $1400^{\circ}$  in  $1\frac{1}{2}$  hr., after which this temperature was maintained for 1 hour.

1. *Nature of the Quartz.*—In these tests, the quartz was introduced in fragments as large as hazel nuts, but most of the fragments decrepitated in the furnace. The density of the samples after firing is shown in Table 5.

TABLE 5.—*Effect of Nature of Quartz on Ultimate Density*

SOURCE OF QUARTZ	DENSITY AFTER HEATING
Pebbles from Central Plateau.....	2.65
Bouchard quartzite.....	2.62
Souigny quartzite (very pure).....	2.60
Urçay quartz.....	2.36
Plat quartz.....	2.35
Souigny quartzite (very impure) ...	2.32
Silex.....	2.30

2. *Size of the Fragments.*—These tests were conducted with the two varieties of quartz which were found the most difficult to transform. Results are given in Table 6.

TABLE 6.—*Effect of Size of Particles on Ultimate Density*

	PEBBLES FROM CENTRAL PLATEAU. FINAL DENSITY	ALLIER QUARTZ (VERY PURE). FINAL DENSITY
Larger pieces .....	2.65	2.60
Between 80 and 200 mesh*	2.64	2.57
Below 200 mesh.....	2.59	2.39

3. *Nature of the Flux.*—These, and all the following tests, were made on pulverized quartz retained between screens having 800 and 4900 openings per sq. cm. (approximately 80 and 200 mesh per linear inch). The temperature, as before, was maintained at  $1400^{\circ}$  for 1 hour. In each case, the amount of flux added was 3 per cent.; the quartz was Fontainebleau sand. The effect of different fluxes on the final density of the quartz is shown in Table 7.

TABLE 7.—*Effect on Ultimate Density of Adding 3 Per Cent. Flux to Fontainebleau Sand*

FLUX	FINAL DENSITY	FLUX	FINAL DENSITY	FLUX	FINAL DENSITY
None	2.60	$\text{Li}_2\text{CO}_3$	2.30	$\text{TiO}_2$	2.56
$\text{Na}_2\text{CO}_3$	2.40	$\text{CaO}$	2.53	$\text{B}_2\text{O}_3$	2.49
$\text{Na}_2\text{SiO}_3$	2.34	$\text{Al}_2\text{O}_3$	2.60	$\text{CaF}_2$	2.51
$\text{NaCl}$	2.45	$\text{Fe}_2\text{O}_3$	2.58	$\text{BaCl}_2$	2.53
Glass	2.32	$\text{Pb}_3\text{O}_4$	2.49	$\text{ZnO}$	2.60

4. *Influence of Proportion of Flux.*—The effect of varying the proportion of two fluxes, silicate of sodium, and lime, is shown in Table 8.

\* Between 800 and 4900 holes per square centimeter.

TABLE 8.—*Effect on Ultimate Density of Varying Proportions of Flux*

Sodium Silicate Flux		Lime Flux	
Per Cent.	Final Density	Per Cent.	Final Density
0.0	2.60	0	2.60
0.5	2.52	3	2.53
1.5	2.45	6	2.46
3.0	2.34		

5. *Influence of Temperature.*—In these experiments, the heating occupied  $1\frac{1}{2}$  hr., the temperature then being maintained at  $1400^{\circ}$  for 1 hr. or for 3 hours. The results are given in Table 9.

TABLE 9.—*Influence of Time of Heating on Density of Quartz*

INGREDIENTS	TIME OF HEATING	FINAL DENSITY
Sand alone.....	1 hr.	2.60
Sand alone .....	3 hr.	2.51
Sand and 3% CaO.....	1 hr.	2.53
Sand and 3% CaO.....	3 hr.	2.46
Sand and 3% NaCl.....	1 hr.	2.51
Sand and 3% NaCl.....	3 hr.	2.38

6. *Temperature of Heating.*—In these tests, the sand was heated alone, or with the addition of 3 per cent. of fluxing material, to temperatures of  $1300^{\circ}$ ,  $1400^{\circ}$ , and  $1500^{\circ}$ . The results are shown in Table 10.

TABLE 10.—*Effect of Temperature on Density of Quartz*

	$1300^{\circ}$ C.	$1400^{\circ}$ C.	$1500^{\circ}$ C.
	Density	Density	Density
Sand alone.....	2.65	2.60	2.44
Sand and 3% $\text{Na}_2\text{SiO}_3$ .....	2.31	2.34	2.32
Sand and 3% $\text{CaF}_2$ .....	2.56	2.51	2.35
Sand and 3% $\text{CaCl}_2$ .....	2.54	2.51	2.35
Sand and 3% $\text{Pb}_3\text{O}_4$ .....	2.53	2.49	2.37

It is reasonable to suppose that the natural impurities of quartzites might explain their relative ease of transformation; chemical analyses, however, have not provided any clear indication. The most frequent impurities are mica, interspersed between the grains, and oxide of iron as very finely divided inclusions in the crystals. The addition of calcium fluoride does not seem to have produced any very energetic reaction. Possibly inclusions of water or of carbon dioxide in certain quartz crystals may exert some influence.

The most obvious conclusion seems to be that quartzites containing chalcedony are transformed at the lowest temperatures. Often, however, when the proportion of this variety of silica is large, the mass falls to powder during calcination and is unable to yield substantial brick.

## MANUFACTURING OPERATIONS

To assist in ascertaining the method of manufacture that will yield the best brick, Table 11 will indicate the lines of relationship.

TABLE 11.—*Essential Conditions and Steps in Manufacture*

CONDITIONS	PROCESSES
1. Nature of quartz	1. Selection of quartz
2. Size and shape of grains	2. Preliminary calcination
3. Nature of fluxes	3. Crushing
4. Proportion of fluxes	4. Addition of fluxes
5. Tempering water	5. Wetting the mixture
6. Uniformity of mixture	6. Mixing
7. Compression of mixture	7. Molding
8. Temperature of firing	8. Drying
9. Duration of firing	9. Firing

*Selection of the Quartz*

1. *Degree of Purity.*—The proportion of basic oxides should not exceed 3 per cent., which is equivalent to 10 per cent. of sulfate, if excessive fusibility is to be avoided. On the other hand, the percentage of impurity should not be below 1.5 per cent. to avoid the necessity for excessively high temperatures in order to cause complete transformation of quartz. An average of 2 per cent. of basic oxides represents good practice throughout the world. The proportion of basic oxides in a number of natural materials used for silica brick is shown in Table 12.

There may be some doubt as to the correctness of this opinion. It is certain that a very pure quartz, mixed with an equally pure lime, does not lend itself well to the manufacture of silica brick, their temperature of firing being below the melting points of the most fusible silicates of lime. On adding iron and alumina to the mixture, which occur naturally in impure quartz, satisfactory results may be obtained. A sufficient quantity of iron is often added by the wear of the crushing apparatus itself. It may be questioned whether brick made with the more difficultly transformed quartz, which are also the most expensive to make, are actually the best product. The high temperature, necessary for firing, at the same time facilitates the transformation of cristobalite into tridymite.

TABLE 12.—*Percentage of Basic Oxides in Certain Siliceous Materials*

	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	OTHERS	TOTAL
Dinas quartzite.....	1.60	0.30	0.40	2.30
Sheffield black ganister.....	0.30	1.50	0.20	2.00
German quartzite (Stella).....	1.50	0.50	0.50	2.50
Souigny quartzite (Allier).....	0.60	1.55	0.40	2.55

2. *Absence of Pulverization During Firing.*—Certain quartzes fall to powder during calcination and naturally cannot yield other than very

ordinary brick; the large grains, the importance of which has been noted above, disappear during this operation. This fault is easily detected by a rapid heating to between  $1500^{\circ}$  and  $1600^{\circ}$ , sufficient to cause transformation of the quartz into silica of low density. This defect often appears to be due to the presence of chalcedony, sometimes accompanied by opal. This last form of silica is difficult to distinguish under the microscope, but its presence can always be recognized by the loss of weight during calcination. It is always advisable, before using a new quartz on a large scale, to subject it to a preliminary calcination at high temperature in order to determine its behavior.

3. *Hardness of the Rock.*—Rocks of great hardness increase the expense of crushing; while, on the other hand, a rock that is too soft makes it difficult to obtain large grains, and especially those of angular shape. For this reason, true quartzites are generally preferred to sandstone, although the latter can be crushed more cheaply. In England, however, the Sheffield ganister, which has a very high reputation for the manufacture of silica brick, is a true sandstone.

Sands are the worst of all natural quartz materials for this purpose, on account of the fineness, the rounded outline, and especially the uniformity in size of their grains. The Fontainebleau sand, for example, contains only grains ranging between 0.1 and 0.3 mm. Sand can well be used, however, for the preparation of the impalpable material, the necessity for which has been indicated above. It would seem possible, nevertheless, to employ it in larger proportions for the manufacture of silica brick. We made briquets containing 75 per cent. Fontainebleau sand, 25 per cent. of impalpable, and 2 per cent. lime; this mixture, after burning at a temperature a little higher than the average, gave a product having an absolute density of 2.32, that is to say, the quartz has been entirely transformed into silica of low density. Compressive strength at ordinary temperatures was 112 kg. per sq. cm., which is ample, and expansion during firing had been 3 per cent., which is a normal amount, but these bricks disintegrated completely at high temperatures on account of the absence of large grains.

4. *Ease of Transformation of Quartz.*—The different varieties of quartz are transformed with varying ease under the application of heat. For example, quartz broken into grains of less than 1 mm. diameter, but remaining on a 200-mesh screen (4900 meshes per sq. cm.), is reduced to a density below 2.40 after 1 hr. of firing, at the following temperatures: Silex,  $1300^{\circ}$ ; ribbon quartzite from Allier,  $1400^{\circ}$ ; pure vein quartz,  $1500^{\circ}$ .

It is not yet possible to say which is the best. It seems well established that silex always yields poor brick; they are light, very porous, and lack rigidity. The expansion of the largest grains is accomplished before fusion of the flux, and therefore before the formation of the

tridymite network. Those varieties of quartz which are most difficult to transform seem to yield the best products, provided the firing is continued long enough to give a complete transformation of the quartz; this high temperature tends to develop the production of tridymite and diminish that of cristobalite. Abundance of cristobalite is the principal cause of fracture, a very serious defect. At present, the preference is generally for quartz which transforms with moderate ease, yielding sufficiently satisfactory products at a moderate cost.

### *Calcination of Quartz*

A preliminary calcination of quartz, now rarely employed, can be given for two different purposes. The first object is simply to weaken the quartz in order to facilitate its crushing. For this purpose a temperature of around 1000° is sufficient, which can be obtained with a moderate expenditure of fuel. Except in the case of very pure hard quartz, this operation seems to have little advantage, the expense of the firing exceeding the economy realized during crushing. Furthermore, quartz weakened in this manner gives rise to rounded fragments, and this practice is tending to disappear.

In the second place, calcination at very high temperature may be adopted in order to produce a transformation of the quartz before it is introduced into the brick-making mixture. This necessitates a large consumption of fuel and complicated heating furnaces. It is essential to perform the heating with gas or with a flame, avoiding direct contact of ashes, in order not to discolor the calcined material. A possible application of this method would be to subject the largest grains of quartz to a partial transformation before introducing them into the brick mixture; this would undoubtedly permit a reduction in temperature and in duration of the firing of the brick.

### *Crushing*

The crushing of quartz is always an expensive operation on account of the hardness of the material; it requires the expenditure of considerable energy and leads to a rapid wear of the crushing apparatus. Roller mills are most commonly applied, the operation being continued until the desired degree of fineness is obtained. This does not seem a rational method, because the relative proportion of the different sizes of material cannot be accurately regulated.

The necessity for a large proportion of impalpable material is one of the clearest results of our experiments, some of which are shown in Table 13. On comparing the results of the first two experiments, for example, one mixture containing 25 per cent. of impalpable obtained in a tube-mill, the other containing 25 per cent. of fine material obtained by screening an ordinary crushed product, it is seen that the compressive strength



at 1600° varies in the proportion of 4 to 1, and at ordinary temperatures in the proportion of 2.5 to 1.

The preparation of impalpable material should be done in a tube-mill, starting with siliceous material already in a fine state, such as the Fontainebleau sand, to which, if necessary, 5 per cent. of burnt clay brick can be added in order to introduce the necessary amount of iron and alumina, such as naturally exists in quartzite. On the other hand, the table shows that the amount of impalpable material should not be too large. Comparing experiments No. 1 and 3, it will be seen that a brick containing

TABLE 13.—*Effect of Varying Fineness of Quartz on Properties of Silica Brick*

No.	Composition of Mixture	Linear Expansion, Per Cent.	Density		Crushing Strength, Kg. per Sq. Cm.		
			Apparent	Actual	At Ordinary Temp.		At 1600° C.
					Dried	Fired	
1	Fresh quartzite, 75 Powdered quartz, 25 Lime, 2	5.2	1.63	2.35	15	165	30
2	Fresh quartzite, 75 Screenings, 25 Lime, 2	5.2	1.63	2.33	10	60	8
3	Fresh quartzite, 25 Powdered quartz, 75 Lime, 2	3.9	1.36	2.35	9	135	10
4	Fresh quartzite, 25 Screenings, 75 Lime, 2	3.9	1.36	2.33	6	52	3
5	Calcined quartzite, 75 Powdered quartz, 25 Lime, 2	3.2	1.57	2.33	10	120	25
6	Calcined quartzite, 25 Powdered quartz, 75 Lime, 2	3.0	1.35	2.34	8	180	15
7	Calcined quartzite, 25 Powdered quartz, 75 Marly clay, 6	5.0	1.40	2.35	9	150	9.5
8	Fresh quartzite, 25 Powdered quartz, 75 Marly clay, 6	5.2	1.43	2.36	9.5	160	6
9	Fresh quartzite, 25 Screenings, 75 Marly clay, 6	5.2	1.43	2.34	7	55	2
10	Fresh quartzite, 75 Powdered quartz, 25 Marly clay, 6	5.0	1.50	2.34	19	110	16
11	Fresh quartzite, 75	3.7	1.60	2.35	...	120	24
12	Powdered quartz, 25	4.1	1.60	2.36	...	150	28
13	Lime, 2	4.5	1.78	2.37	...	250	25

25 per cent. of impalpable has a crushing resistance at high temperature three times that of a brick containing 75 per cent. of impalpable; we would therefore recommend a proportion of 25 per cent. of impalpable material, passing the 200-mesh screen.

To obtain the larger grains there is no reason for using roller mills, and it is preferable to use some form of cylindrical crusher, requiring much less mechanical energy, and also yielding grains having a lamellar shape, which is most advantageous for the compactness of the brick.

If roller mills must be used, however, it is necessary to give them a sufficient weight, 5 tons at least, in order to enable them easily to crush fragments of quartz of the size usually delivered by the jaw crusher. If the roller mills are too light, they roll over the grains without crushing them, increasing greatly the cost of power and repairs, while also introducing particles of iron into the mixture, which later give rise to brown stains on the brick, after firing.

#### *Addition of Fluxes*

Lime is the only flux regularly employed by manufacturers of silica brick; the proportion is generally between 1 and 2 per cent. M. Bied has proposed to add to the lime either oxide of iron or alkali. The advantage of a flux containing oxide of iron is that silica is only slightly soluble in it at high temperatures, and therefore the tridymite network is less rapidly destroyed than in other fluxes. In Martin furnaces, the bricks of the roof are often impregnated with oxide of iron to a depth of 10 cm. without seriously diminishing their resistance to heat.

The presence of alkalies greatly facilitates the transformation of quartz into silica of low density, especially into tridymite. Alkalies can be introduced in the form of alkaline clay, such as the majority of marly clays, glauconite, and the clays of Fresnes and Salerne. On the other hand, alkalies have the disadvantage of exerting an energetic soluble action on the tridymite network. Our test No. 10, Table 13, was very satisfactory, nevertheless.

Lime alone does not seem a sufficient flux, for the pure silicates of lime do not fuse until they reach temperatures above those obtained in the firing of silica brick. The quartzites ordinarily employed, however, contain 2 per cent. of alumina and iron, which, with the lime, yield silico-aluminates and silico-ferrites, fusible at about 1200°. When very pure quartz is to be employed, it seems indispensable to add a certain proportion of oxide of iron. Silica brick are often made without any addition of flux, the mica, after crushing, possessing enough adhesive power to give the dried brick a suitable stiffness, and sufficient fusing power to permit recrystallization of silica during the firing.

The chemical composition of the fluxes is not the only important point; it is necessary to reduce them to a sufficiently fine state of division to

allow them to mix intimately with the silica; this is the more important in proportion to the amount of impalpable silica in the mixture. Well slacked, fat lime and natural clays are generally sufficiently fine; when well-burned hydraulic limes are used, which are always granular, it is necessary to pass them through a tube-mill with the impalpable silica. The best method to insure that a fat lime shall be sufficiently finely disseminated is to slack it in three or four times its weight of boiling water and then use the milk of lime without allowing it to become dry, in order to avoid agglomeration. It is not necessary to have all the water boiling at the start; the operation can be begun with a little boiling water, after which increasing quantities of lime and cold water can be introduced, as the heat of reaction develops.

#### *Wetting the Mixture*

The proportion of water added for the purpose of making the mixture workable should be enough to permit the brick to be carried to the dry house without danger of deformation. The quantity may vary from 8 to 16 per cent. according to the proportion of impalpable material; obviously, the larger the proportion of extremely fine grains the more water is required. Furthermore, the impalpable material so increases the compactness of the brick as to allow a larger proportion of water to be used without making the brick too soft. It sometimes happens, when introducing a large amount of impalpable material, that the operator forgets to increase the proportion of water; thereupon the brick, when subjected to firing, break in planes perpendicular to the direction of compression, owing simply to a lack of water.

#### *Mixing*

Mixing, for the purpose of distributing the flux uniformly throughout the siliceous mass, is the more necessary according to the proportions of fine material. We have not yet found a perfectly satisfactory process for controlling the distribution of lime throughout the mixture, although this is a very important factor determining the quality of the brick. Inasmuch as the mixing operation is not very expensive, it would be much better to increase the length of the mixing process, even beyond what would appear to be strictly necessary. The operation is generally conducted in light mills revolving rapidly.

On this subject, we would suggest the possibility of using, for mixing, the impact mills, which are used for the preparation of molding sand at foundries. We would also indicate, with some reserve, the possible advantage of introducing the water gradually. When it contains only 5 per cent. of water, the mass mixes readily and remains sandy; the additional water can then be introduced at the end of the operation, when it will distribute itself immediately and uniformly.

### *Molding*

Molding of the bricks is most often performed by hand, although there is some doubt as to whether this process is better than the use of a molding press. It permits, possibly, a more regular distribution of the mixture in the molds, and yields brick which, at 1600°, possess the same mechanical strength as machine-pressed brick. In the case of badly fired brick, the final expansion, which occurs after the bricks are in the furnace roof, has better opportunity to relieve itself in the spaces of a very porous brick and thus produces much less external pressure. On the other hand, pressed brick, which are always dense and have greater strength at ordinary temperature, are much less permeable to the slag; this is important, as this permeability is an important factor in the destruction of brick. It would seem, finally, that for careful manufacture, high molding pressures are preferable, although for second-quality brick hand molding may be perfectly suitable.

### *Drying*

The molded brick must be dried before they are introduced into the kiln because they would otherwise be too soft to permit them to be piled one on another; the abrupt application of heat, furthermore, would cause them to burst or at least crack by too rapid expelling of excessive water vapor.

The drying operation presents no difficulties and requires no special precaution. It is possible, immediately after molding, to put the bricks into a heated stove and dry them in a few hours. In this respect, silica bricks differ from clay bricks in that drying does not produce any contraction. With bricks containing a large amount of impalpable silica, the operation may require more care, but is never difficult.

### *Firing*

Firing is the most important feature in the manufacture of silica brick, and also the most expensive; unfortunately the best conditions for firing are not yet fully understood. Tests are difficult to make on account of the length of firing and the dimensions of the furnace in which firing is done; firing often lasts for 20 days and may take place in a furnace holding 200 to 300 tons of brick at once.

The maximum temperature of firing is often considerably exaggerated. We often hear of firing temperatures of 1500° and even 1600°, but we do not believe that any silica brick are ever actually fired at temperatures exceeding 1400°, and base this belief on the two following facts: In the most intensive firing, the heat is generally limited to Seger cones No. 16 to 18, corresponding nominally to temperatures of 1450° and 1490°; numerous experiments, however, have shown us that in ceramic furnaces, in which the heat is maintained for a long time, Seger cones melt at

temperatures between  $70^{\circ}$  and  $100^{\circ}$  lower than in calibration tests, which are always conducted very rapidly. Furthermore, on examining the expansion of commercial brick, produced by subjecting to a second firing, we have determined that they all began to expand rapidly upon reaching a temperature of  $1400^{\circ}$ , which proves that they had not undergone this temperature in the first firing.

We believe, therefore, that a temperature of  $1400^{\circ}$  is enough for firing, provided it is maintained for a sufficiently great length of time. In any case, we do not believe that there is any advantage in exceeding a temperature of  $1450^{\circ}$ , even with the most difficultly transformed quartz. Our experiments, mentioned above, show that the Fontainebleau sand,



FIG. 11.

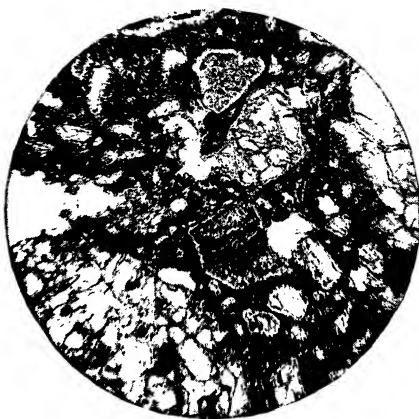


FIG. 12.

FIG. 11.—SILICA BRICK MADE FROM SOUVIGNY QUARTZITE. CIRCULAR CLEAVAGES CHARACTERISTIC OF CRISTOBALITE, IN LARGE GRAINS OF QUARTZITE ENTIRELY TRANSFORMED BY THE FIRING. NATURAL LIGHT; MAGNIFICATION,  $\times 34$ .

FIG. 12.—SILICA BRICK MADE OF SOUVIGNY QUARTZITE, SHOWING FRAGMENTS OF QUARTZ NOT TRANSFORMED BY FIRING. AROUND THE LARGE GRAINS IS A BORDER 0.01 MM. DEEP PRODUCED BY THE ATTACK OF THE FLUX. NATURAL LIGHT; MAGNIFICATION,  $\times 34$ .

which is particularly difficult to transform, in the presence of lime is reduced to a density of 2.46 after 3 hr. of heating at  $1400^{\circ}$ .

Finally, numerous tests of mixtures containing Fontainebleau sand fired in industrial furnaces have yielded densities between 2.32 and 2.36. As for the large grains of quartz, the transformation is less rapid and it would obviously be advantageous to introduce these, if this could be done, in the condition of quartz already transformed.

In addition to the temperature, it is necessary to take into account the length of time during which the maximum temperature is maintained, and also the rapidity with which the heating is conducted. In case of too rapid a heating, the brick expands enormously, the direct transformation of the grains having preceded the formation of the network. From the

theoretical point of view, it would seem that the best condition of firing would be to raise the bricks as rapidly as possible to the temperature at which the large quartz grains begin to transform directly, but slowly; this would give the network of tridymite an opportunity to develop more rapidly than the isolated grains transform, which is indispensable in order to limit expansion. This temperature would then be maintained a sufficiently long time to allow the transformation of large grains to be completed.

We should mention an absolutely contrary theory maintained by certain manufacturers on account of its economical advantages. This involves firing at a very low temperature, in order to form the indispen-

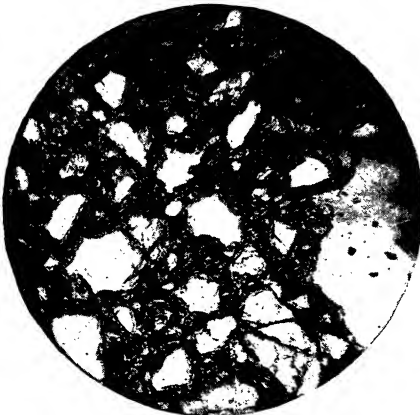


FIG. 13.

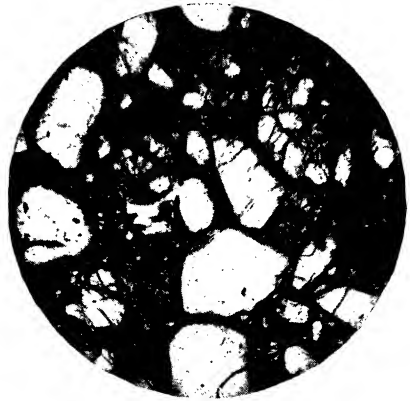


FIG. 14.

FIG. 13.—INSUFFICIENTLY FIRED ENGLISH BRICK, CONTAINING UNTRANSFORMED GRAINS OF QUARTZ, ALTHOUGH BORDERED TO A DEPTH OF 0.01 MM. BY ATTACK OF THE FLUX. NATURAL LIGHT; MAGNIFICATION,  $\times 34$ .

FIG. 14.—INSUFFICIENTLY BURNED GERMAN BRICK, CONTAINING ROUNDED GRAINS OF QUARTZ, INDICATING THE USE OF A NATURAL SAND. NATURAL LIGHT; MAGNIFICATION,  $\times 34$ .

sable network, but allowing the grains of quartz to remain untransformed; the final heating is afterward finished in the steel furnace. This process has the advantage of not introducing cristobalite into the brick and thereby diminishing the danger of fracture; on the other hand, at high temperatures, brick of this character would be subject to considerable expansion, leading to a warping of the furnace roof. If, however, the proportion of impalpable silica has been sufficient, and if the firing has been sufficiently prolonged, the tridymite network may perhaps be sufficiently solid to offset the danger of expansion.

We should also mention a third theory, upheld by certain American authors; well burned bricks are good; slightly burned bricks are mediocre; but medium burned bricks are detestable. Such brick are composed principally of cristobalite and disintegrate into large fragments by fracturing.

A Symposium on the Conservation of Tin

(Milwaukee Meeting, October, 1918)

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Bronze Bearing Metals

G. H. CLAMER,\* Philadelphia, Pa.—Unfortunately, prior to the war no serious attention was given to the conservation of tin, notwithstanding that this country is practically dependent upon outside sources for its entire tin supply.

Tin is of vital importance in many industries, but it is surprising how many and how excellent are its substitutes when we become acquainted with them. Tin has always been a relatively high-priced metal, and it is part of the human attitude to associate high prices with high standards; it is not until the price of a commodity becomes well-nigh prohibitive that we hunt for substitutes, because the idea of substitution seems always to involve an assumption that the substitute must necessarily be inferior.

The history of the development of bearing bronzes is a striking example of this policy. In the early days, copper-tin alloys were almost universally used, the idea then being prevalent, which is still held by many, that a bearing to resist wear must be hard, and the harder the better. The favorite bronze bearing contained 90 per cent. copper and 10 per cent. tin; frequently, in service which was considered severe, even higher proportions of tin were used. Such hard alloys have great resistance to compression, but as a rule they had a very wide factor of safety in this

\* First Vice-president and Secretary, Ajax Metal Co.

respect. Such bearings, because of their inability to adjust their surfaces to slight irregularities in the journal, or to foreign bodies, immediately begin to cut, and heating results. With a slight rise in temperature, the film of lubricant becomes thinner, and further cutting follows, if not actual gripment of the bearing with the journal.

Many years ago, Dick, of England, appreciating the advantage to be derived from a slight plasticity in a bearing, added some lead to the then standard bearing metal, not substituting lead for tin but reducing the copper, and produced the alloy which has long held favor as a bearing metal, *i.e.*, copper, 80; tin, 10; lead, 10 per cent. Dick's alloy also contained some phosphorus, but the main point is that this was the first step toward the production of bronze alloys having a plastic nature. Lead does not unite to form an alloy with copper, but remains mechanically mixed, so that the structure of the alloy is that of a hard matrix with the soft metal imbedded therein.

It was not until several years later that tests were conducted on the Pennsylvania Railroad, under the direction of Dr. C. B. Dudley, who investigated the copper-tin-lead series within certain limits of the 80-10-10 alloy; he studied not only the alloys containing lead above 10 per cent. in which copper was replaced by lead, but also in which tin was replaced by lead. His conclusions, which have since become firmly established, are: (1) The rate of wear diminishes with increase of lead in the alloy. (2) The rate of wear diminishes with decrease of tin in the alloy. Fortunately, the alloy containing least tin and highest lead exhibits least tendency, in service, to give trouble from heating.

Notwithstanding the decided merit of copper-tin bearings containing lead, prejudice was strongly against them, simply because lead is a low-priced metal. It was even intimated that such alloys were frauds, should be considered such, and dealt with accordingly.

I have mentioned Dr. Dudley's discoveries because it was due to his findings that we instigated research work, now 20 years ago, which has led to the production of alloys still higher in lead and lower in tin than those which he was able to produce. He experienced foundry difficulties which apparently limited his maximum-lead alloy to 77 copper, 8 tin, and 15 lead. This was called Experiment B alloy, and has since been widely known as "Ex. B metal."

Having due regard to the raw materials used, and by following good foundry practice, we have been able to produce alloys carrying 5 per cent. of tin and as much as 30 per cent. of lead which would show no segregation of lead, even if cast into large bearings. By this I mean that such bearings will show no indication of metallic lead upon any surfaces. Lead being only mechanically held in the alloy, it is prevented from segregating only by the quick setting of the matrix of copper and tin. As a certain interval must necessarily occur between the time when the metal



enters the mold and the time when it solidifies, the lead always shows some tendency, owing to its high specific gravity, to liquefy toward the bottom of the casting. In bearings made of the proper raw materials, and correctly handled, the difference in the proportion of lead is not usually over a fraction of 1 per cent., or at most 2 or 3 per cent., between the top and the bottom of a casting, even if this be a fairly large one, and made of the 30-per cent. lead alloy.

I do not wish to repeat here data which I have given in previous papers<sup>1</sup> but I do wish to set forth the position which the high-lead and low-tin alloys developed by us have attained. When these alloys were first produced they were backed only by laboratory tests and by the predictions of Dr. Dudley that, if such alloys could be commercially produced, the law which he established would no doubt apply also to alloys higher in lead and lower in tin than those which he had developed. It is now possible for me to review 18 years' experience with the manufacture and service of such bearings. I must confess that in our enthusiasm over the valuable properties of these alloys, we were led at times to overstep the mark and place such bearings in service where the loads or the impacts were too great.

The first requisite of a bearing is that it shall be sufficiently hard to support its load or to resist the impacts to which it may be subjected, and the relation of tin to lead must be controlled by this requirement. We have sometimes made mistakes in recommending the copper 65, tin 5, lead 30 alloy for certain mill bearings; this did not have sufficient resistance to compression, and failed for that reason. When the copper 73, tin 7, lead 20 alloy was substituted, the bearings exhibited no deformation and performed far better than the 80-10-10 alloy previously used. We have also noted the failure of the 73-7-20 alloy on rod bearings of very heavy locomotives. Locomotive rod bearings are subjected to severe impacts and it is necessary therefore to use an alloy of fairly high compressive strength. Although the above alloy performs satisfactorily on light locomotives, on the rod bearings of heavy locomotives it is necessary to use either the 80-10-10 alloy or the same alloy to which has been added approximately 1 per cent. of phosphorus. The size of these bearings, and hence their bearing surface, is narrowly limited by necessities of construction; otherwise these harder alloys would not be essential for this purpose. Phosphorus greatly increases the compressive strength of such an alloy, and is for this reason a possible factor for conserving

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<sup>1</sup> For example: A Study of Alloys Suitable for Bearing Purposes. *Journal, Franklin Institute* (July, 1903) 166, 49. History and Development of the Alloy Practice in the United States as Applied to Railway Bearings. *Proceedings, American Society for Testing Materials* (1907) 7, 302. Effect of Changes in the Composition of Alloys Used by the American Railways for Car-journal Bearings. *Transactions, American Institute of Metals* (1915) 9, 241.

tin. At the present prices of tin and phosphorus there is little choice; an alloy with 8 per cent. of tin and 1 per cent. of phosphorus will have compressive strength approximately equivalent to the alloy having 10 per cent. tin. Experience, thus, has demonstrated that alloys containing as little as 5 or even 4 per cent. tin and as high as 30 per cent. lead, can be used in railroad service for car bearings. They have become the standard of the United States Railroad Administration for car-journal bearings called for under their Specifications R-71, Grade A. Such an alloy is also included in the specifications covering locomotive bearings designated as Specification R-72, Soft Bronze.

In my judgment, the specifications of the Railroad Administration covering locomotive bearing-metals are very satisfactory, except that the use of soft bronze should be extended to cover driving brasses, and engine and trailer-truck bearings. Before the railroads of the United States came under Government control, copper alloys with low-tin and high-lead contents had become the standard specifications of several of the large car companies, and were very extensively used on the largest railroad systems. Outside of the railroad field they had also been widely recognized and used. The advantage of using the smallest possible amount of tin consistent with the load requirements is now so well understood that there is but little opportunity for an important conservation of tin in this field.

Let us next consider the possibilities of substituting some other metal for a part or all of the tin in a copper-tin-lead alloy, or of substituting alloys of an entirely different type.

The first metal that presents itself as a substitute for tin is antimony. Antimony combines readily with copper and with lead, and has the property of adding hardness. Unfortunately, however, the hardening effect of antimony is obtained with the sacrifice of ductility. We have found it possible to make alloys carrying as high as 30 per cent. of lead with 3 per cent. of tin and 2 per cent. of antimony. We have also made alloys of 65 copper, 30 lead, 2 tin, and 3 antimony, and have also replaced the 5 per cent. of tin in this alloy entirely with antimony. Car bearings  $4\frac{1}{4}$  by 8-in. size, made from the same pattern on molding machines and subjected to a breaking stress applied longitudinally at the middle of the back of the bearing and throughout its entire length, broke at the following average loads: with 2 per cent. antimony substitution, 60,000 lb.; with 3 per cent. substitution, 62,000 lb.; with total substitution, 52,000 lb.; as compared with a breaking load of 67,000 lb. for the alloy of copper 65, tin 5, lead 30. The castings produced with each of the three above-mentioned alloys are not so satisfactory as those made with the straight-tin alloys, being more or less rough, and showing slight globules of lead on the surface. It has been found that a certain amount of nickel can be used for replacing tin with very satisfactory results. The castings

produced when zinc is substituted for a certain amount of tin are decidedly unsatisfactory. The substitution of aluminum for tin is entirely impractical, and such castings are worthless. This does not, however, exhaust all the possibilities of substituting other metals for tin in the copper-tin-lead alloys, but it is my opinion that the substitution of any other metals, in those alloys, can be made only by sacrificing the quality of the alloy.

The possibility of substituting alloys of an entirely different type presents an attractive field for research. The copper-tin-lead alloy has attained its position as the most desirable bronze bearing alloy, but this does not mean that some other alloy may not be found which may give equally good or better results. In the search for such a substitute alloy it should be borne in mind that a bearing metal should possess the following properties:

(1) It should be sufficiently rigid to support the load or resist the impact, but yet not so brittle that it will easily crack.

(2) It should have as great a yielding or plastic nature as is consistent with its ability to support the load or resist the impact without deformation of the bearing as a whole.

(3) The ideal structure combines a hard matrix to support the load and a softer metal or alloy contained within such matrix, to permit the bearing surface to adjust itself to irregularities of service.

(4) It should be easy to handle in the foundry and machine shop.

(5) It should be capable of being remelted without deterioration.

(6) For use in babbitt-lined bearings, it should be capable of being tinned, so that the babbitt can be applied thereto.

(7) It should have good heat conductivity in order to dissipate the heat generated by friction.

#### **Pennsylvania Railroad Anti-friction and Bell Metals**

F. M. WARING,\* Altoona, Pa.—The necessity for conserving tin has recently been very forcibly brought to the attention of all consumers, and efforts are now being made to reduce the tin content in certain alloys or to substitute other alloys not containing tin.

The approximate composition of the non-ferrous alloys in general use on the Pennsylvania Railroad are given in the accompanying table.

Phosphor bronze is used principally for rod bushings, main-rod brasses, and crosshead shoes.

Ex. B bronze is used to a small extent for backs of car and coach bearings, but the majority of these are now made of the car-journal bronze, which contains, on the average, about 5 per cent. tin.

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\* Engineer of Tests, The Pennsylvania Railroad Co.

*Composition of Non-ferrous Alloys Used by Pennsylvania Railroad*

	Copper	Tin	Lead	Phos.	Antimony	Zinc
Phosphor bronze, Spec. 32-C.....	79.70	10.00	9.50	0 80		
Ex. B bronze, Spec. 141.....	76.75	8.00	15.00	0.25		
Car-journal bronze (a).....	(b)	(c)	(d) (e)	.....	.....	(f)
Special high-lead bronze, Spec. 59.	70.00	5 00	25.00			
Lining metal, Spec. 57.....	.....	.....	87.00	.....	13 00	
Dandelion metal.....	.....	10 00	72.00	.....	18 00	
Bell metal.....	83 $\frac{1}{3}$	16 $\frac{2}{3}$				
Babbitt, tin-base.....	3.70	88.90	.....	.....	7.40	
Babbitt for motor bearings.....	1.00	50.00	38.50	.....	10 50	

(a) Sum of Cu, Pb, Sn, and Zn, not less than 99. (b) Not less than 71. (c) Not less than 4. (d) Not less than 13. (e) Not more than 20. (f) Not more than 3.

Car-journal bronze is used for making car and coach bearing backs at the Altoona brass foundry, by melting down old backs after removing the linings and making the necessary addition of new metal to bring the composition within the limits given in the table. No new tin is added in making this alloy.

Special high-lead bronze is used principally for locomotive driving-box shells, which are not lined.

The lead-base lining for car-journal bearings was formerly made up in our foundry from lining metal melted off from old bearings and brought up to specification requirements by the addition of such new metal as might be necessary. Some tin was unavoidably introduced from the old bearings, but the amount allowed in the metal was limited to 2 per cent. Lately we have been using this old lining metal in the preparation of the lead-base dandelion metal babbitt, thus making use of the contained tin in order to reduce the amount of new tin which it was necessary to add to this metal. The journal-lining metal is then made from lead and antimony without the addition of any tin.

Lead-base dandelion metal babbitt, containing about 10 per cent. tin, is used for lining crosshead shoes and also for lining engine-truck and trailer bearings, as well as for hub liners, in place of phosphor bronze, on freight locomotives. This metal has replaced a large amount of tin and tin-base babbitt formerly used.

Bell metal is used exclusively for making locomotive bells, and during 1917 about 42,800 lb. of castings were made, involving the use of a little over 7000 lb. of tin.

Tin-base babbitt metal (88.9 tin, 3.7 copper, 7.4 antimony) is used for a number of miscellaneous purposes in the shops, but its use has been

greatly restricted and every effort is being made to do away with it where possible, and to substitute a lead-base babbitt or a babbitt with 50 per cent. tin.

The amount of solder having the composition 50 lead, 50 tin, used by the Pennsylvania Lines East, during 1917, was approximately 100,000 lb., but there is reason to believe that a large portion of this can be replaced by a 60-lead, 40-tin solder with satisfactory results, and instructions have been issued to this effect.

In regard to the quantity of new tin used, it is not possible to give the amount, except approximately and from calculations based on the 1917 consumption of bearing metals by the Pennsylvania Lines East only, it is estimated that about 770,000 lb. of new tin were required in a total of about 21,380,000 lb. of all kinds of bearing metals turned out by the foundry or purchased in the market.

No change has been made in the specifications for bearing metals for some years, as the metals used have been satisfactory. A large proportion of the bearing metals are made up from old material re-melted and brought to standard composition by some addition of new metal, and every effort is being made to utilize old material to the best advantage and reduce the amount of new metal of all kinds purchased. For a number of years no tin has been used in the lining metal of either passenger or freight car journal-bearings, except such small amounts as come in from re-melting old linings. No change has been made in phosphor bronze used for rod bushings, as we should expect some trouble from bushings pounding out of shape if a phosphor bronze were used which contained less tin or more lead than the present specifications call for. In this, as well as in the case of all other bearing metals, we expect to use our utmost endeavors to economize and to substitute for tin wherever possible.

### The Tin-plate Industry

D. M. BUCK,\* Pittsburg, Pa.—During the first 5 months of 1918, approximately 11,000,000 lb. per month of pig tin were consumed in the United States. Solder, bearing metals, bronzes, etc. used about 5,500,000 lb.; collapsible tubes a little more than 250,000 lb.; tin-foil about 500,000 lb.; and the tin- and terne-plate industry somewhat less than 5,000,000 lb. In an effort to reduce this consumption and thus conserve our tin supplies, several methods of procedure suggest themselves:

1. Salvage. The most careful and systematic collection and re-use of tin and tin-bearing materials is economically important, in that we thus secure the maximum benefits from our available supplies.

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\* Metallurgical Engineer, American Sheet and Tin Plate Co.

2. Substitution of other materials for tin. While tin, on account of its low melting point, softness, malleability, non-toxicity, etc., is peculiarly adapted for many uses, nevertheless it may seem desirable, during times of temporary stringency at least, to substitute for tin and for tin-bearing materials some other substances which may answer our purposes, though perhaps not possessing all of the desirable qualities of tin. It is conceivable that research in this connection may develop entirely satisfactory substitutes which may permanently replace tin for certain purposes.

3. Curtailment, for the time being, of certain lines of manufacture, not absolutely essential to the prosecution of the war.

Efforts along all of the above-mentioned lines are being made in practically all tin-consuming industries, and much progress has been made. In considering the details of tin conservation, it is my intention to confine myself to a brief discussion of this subject as related to the tin- and terne-plate industry.

Terne-plate is a mild-steel sheet coated with an alloy of tin and lead (approximately 25 per cent. tin and 75 per cent. lead). Its chief uses, in normal times, are for roofing, gasoline and oil tanks, and for stamping into various forms. Manufacturers of this material have almost entirely discontinued its manufacture, except to supply the urgent needs of the Government for war purposes.

It has been customary to use a small amount of tin with the spelter in the galvanizing pots in the manufacture of galvanized sheets. It has been found that, by a sacrifice of no other quality than appearance, this tin could be omitted, and the practice has been largely discontinued—entirely so in the concern with which the writer is connected.

Tin-plate consists of thinly rolled mild-steel sheets coated with pure tin, and its chief use is in the canned-food industry. The Government has requested that the manufacturers of this product give absolute priority to orders covering material to be used for the manufacture of plate for cans to contain perishable foods. The manufacturer has, of course, complied with this request and the conditions of the markets have been such that almost the entire capacity of the country has been utilized for such material, and for other direct and indirect Government needs.

Several grades of tin-plate are regularly manufactured, differing only in thickness of the tin coating. While, for some few uses, the heavier coated sheets are desirable and necessary, it is a fact that the most lightly coated sheets are entirely suitable for a very large percentage of these requirements. It is in this connection that the consumer can materially aid in the saving of tin during the present stringency, and also prevent a serious economic waste in normal times, by not specifying a heavier coated plate than his requirements justify.

For years it was believed by certain canners, manufacturers, and dealers in canned goods, that a heavy tin coating was necessary on food containers. This opinion was endorsed by food officials and chemists, and attempts have been made in Congress to regulate the weight of tin coating. Since the literature on the subject gave no definite information, a committee was formed several years ago, consisting of representatives of the American Sheet and Tin Plate Co., American Can Co., and the National Canners' Association. Two representatives of the Bureau of Chemistry, Department of Agriculture, also participated in the work. This committee prepared seven lots of tin plate with the following average coatings, expressed in pounds of tin per base box (112 sheets, 14 by 20 in.).

	POUND		POUND
A.....	0.9	E.....	1.8
B.....	1.1	F.....	2.1
C.....	1.3	G.....	3.0
D.....	1.5		

Cans were made from these plates in the usual way, and various food products were packed under the supervision of the committee, in regular canning plants. Approximately 60,000 cans, in all, were packed with the following foods:

Apples (3 packs)	Milk (evaporated)
String beans	Peas
Cider	Pumpkin (3 packs)
Clam juice	Tomatoes (3 packs)
Corn (3 packs)	Tuna
Milk (condensed)	Salmon

The cans and contents were inspected and analyzed from time to time, throughout a period of about 18 months after filling the cans. In this work more than 40,000 samples were analyzed chemically. I quote from the general conclusions of this committee as embodied in their report:<sup>2</sup>

The most significant fact established by this entire investigation is that, aside from the external appearance of the cans, none of the difficulties encountered in the twenty experimental packs of twelve representative foods in plain cans was taken care of or eliminated by heavy tin coatings. The luster and the resistance to rusting increase somewhat with increased weights of coating. In other respects, with the exception of some instances in the classes of foods that have a tendency to perforate, the conclusion from this work is that the value of different weights of tin coating on food containers is for all practical purposes the same with average weights of from one to three pounds of tin per base box.

<sup>2</sup> "Relative Value of Different Weights of Tin Coatings on Canned Food Containers." Report of an investigation by a technical committee representing the National Canners Association, the American Sheet and Tin Plate Co., and the American Can Co. Published by National Canners Association, Washington, D. C., 1917.

I bring this investigation to your attention to emphasize the needless waste attendant upon the use of tin plate with an unnecessarily heavy tin coating. With our present knowledge, we are unable commercially to produce coatings as light as the lower weights used in this test. If, however, future research should develop means to this end, the resultant product would meet all practical requirements, and a very considerable saving in pig tin would result.

## DISCUSSION

G. H. CLAMER.—The National Canners' Association is studying the action of fruit juices, etc., on solders. Of course these tests will take quite a long time, and we hope by that time the war will be over and there will be no need for conserving tin for war purposes. Germany for a great many years has prohibited the use of more than 37 per cent. of lead in alloys used in contact with food products. The eutectic composition is 37 per cent. of lead and 63 per cent. of tin; alloys containing more than that percentage of lead have some free lead.

J. W. RICHARDS,\* South Bethlehem, Pa.—A great deal of conservation can result from the packing of dry foods in cartons and in fiber packages instead of using sheet tin. Many of the boxes used for packing things other than food are frequently made of sheet tin because it prints and lithographs well; these could be made of sheet iron coated with copper or some other substitute metal which prints equally well.

### The Aluminum Bronze Industry

W. M. CORSE,† Mansfield, Ohio.—The conservation of tin, in view of the shipping situation, is one of great importance. Several methods of conservation can be employed:

1. Reduction of the amount of tin in an alloy or compound.
2. Substitution of an entirely different metal or compound for tin.
3. A combination of the first and second methods.

The second method is the one that I wish to discuss.

Metallic aluminum has been known for a long time, and its use in copper alloys was discovered about 1855 by Lord Percy. The high cost of production of metallic aluminum retarded its commercial development, and it was not until the discovery of the electrochemical processes for its production that it came to be known as a common metal.

I have been particularly interested, for the past few years, in working with the alloy known as aluminum bronze, which is usually composed of approximately 90 parts of copper and 10 parts of aluminum, by weight.

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† Manufacturing Engineer, Ohio Brass Co.



This alloy has many properties similar to the copper-tin bronzes, and it has been of interest to find just where the copper-aluminum bronzes could be substituted for the copper-tin bronzes, and in that way conserve the use of metallic tin.

Copper-aluminum bronzes have practically double the tensile strength of tin bronzes, so that a smaller cross-section frequently can be adopted, with the same mechanical result. Their resistance to shock is superior to that of the copper-tin bronzes, and their resistance to wear is, in some cases superior, and in some cases practically equal. Consequently,

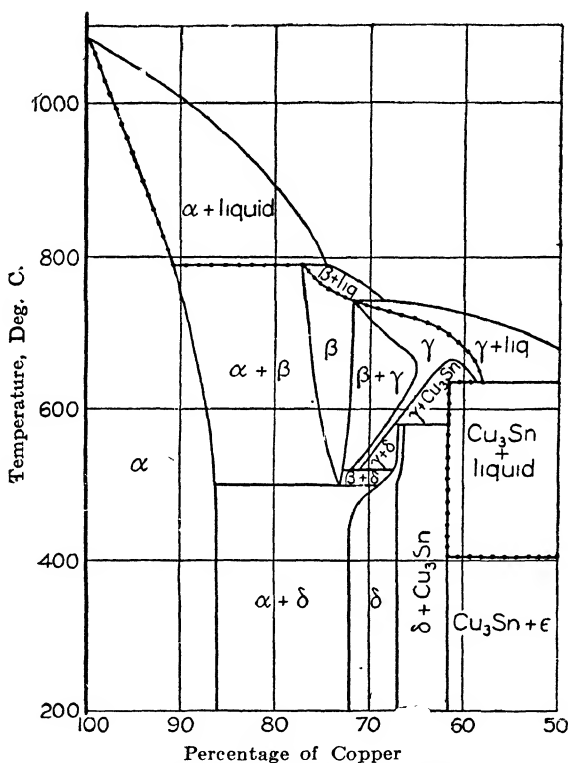


FIG 1.—THERMAL EQUILIBRIUM OR COOLING DIAGRAM OF COPPER-TIN ALLOYS.

for many mechanical uses, where a hard bronze is desired to replace one containing 10 to 11 per cent. of tin, for example, an aluminum bronze of about the composition mentioned will be found worth investigation. Undoubtedly no two alloys possess exactly the same properties, and when a substitution of one for the other is desirable, it is necessary to work out special methods of handling the substitute in order to get practically the same results.

As is frequently the case in such work, special properties are found to be superior to those of the metal originally used, and other properties

are discovered to be not so good. As a particular instance of the substitution of aluminum bronze for phosphor bronze, I would cite its use in worm gearing. The following tables and curves taken from my paper<sup>3</sup> on this subject before the Society of Automotive Engineers will give an idea of the different properties.

TABLE 1.—*Physical Properties of Phosphor Bronze*

(Composed of 88.7 parts of copper, 11 parts of tin, and 0.3 parts of phosphorus)

Ultimate tensile strength, lb. per sq. in.....	35,000–40,000
Yield point, lb. per sq. in.....	22,000–25,000
Elongation in 2 in., per cent.....	6–10
Reduction of area, per cent.....	7–9
Specific gravity at 20° C.....	8.5
Brinell hardness number (500 kg. load for 30 sec.).....	75–85
Pattern maker's allowance for shrinkage, in. per ft.....	0 125
Weight per cu. in., lb.....	0.31
Compression, elastic limit, lb. per sq. in.....	16,000
Coefficient of friction.....	0.0040
Modulus of elasticity.....	12,000,000 to 14,000,000
Resistance to impact, Fremont notched-bar test (fractured section 7 by 10 mm.), kg.-meters.....	2 to 4
Endurance of alternating impact, Landgraf-Turner or Arnold test, alternations.....	150 to 300
Resistance to shear by impact, McAdam machine, ft.-lb.....	300 to 450

Aluminum bronze containing 10 per cent. aluminum and 1 per cent. of iron has the physical properties shown in Table 2.

TABLE 2.—*Physical Properties of Aluminum Bronze*

(Containing 10 per cent. of aluminum, and 1 per cent. of iron.)

Ultimate tensile strength, lb. per sq. in.....	65,000–80,000
Yield point, lb. per sq. in.....	23,000–28,000
Elongation in 2 in., per cent.....	20–30
Reduction of area, per cent.....	21–29
Specific gravity at 20° C.....	7.5
Brinell hardness number (500 kg. load for 30 sec.).....	92–100
Pattern maker's allowance for shrinkage, in. per ft.....	0.22
Weight per cu. in., lb.....	0.27
Compression, elastic limit, lb. per sq. in.....	19,000
Coefficient of friction.....	0.0025
Modulus of elasticity.....	15,000,000–18,000,000
Resistance to impact, Fremont notched-bar test (fractured section 7 by 10 mm.) kg.-meters.....	7 to 10
Endurance of alternating impact, Landgraf-Turner or Arnold test, alternations.....	3,500 to 5,500
Resistance to shear by impact, McAdam machine, ft.-lb.....	750 to 850

<sup>3</sup> W. M. Corse: Worm Gear Bronzes. *Journal, Society of Automotive Engineers*, April, 1918.

It is of interest to note that aluminum bronze would undoubtedly have been substituted for phosphor bronze before this had the manufacturing difficulties with the former been surmounted. Aluminum bronze, when cast in the foundry, presents about as difficult a problem as I have ever seen. It is very sensitive to gas absorption and must be handled extremely carefully to insure good castings. It is similar, from a foundryman's standpoint, to manganese bronze, in that it requires large risers and careful pouring to insure clean castings. Several years' work on this alloy has demonstrated conclusively that it is perfectly

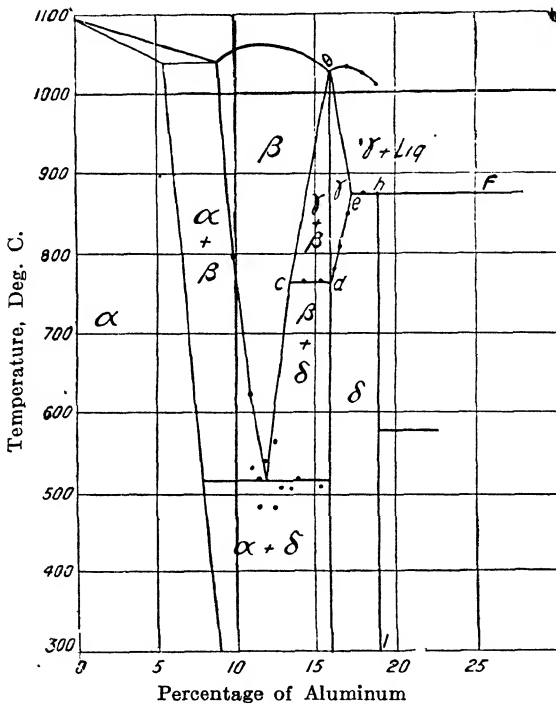


FIG. 2.—THERMAL EQUILIBRIUM OR COOLING DIAGRAM OF COPPER-ALUMINUM ALLOYS.

possible to make as large a percentage of good castings from it as from any other non-ferrous alloy. It seems to me, therefore, that its use should be increased, particularly in view of the shortage of tin at the present time, and undoubtedly new fields will be opened up as its various properties are better known.

One feature that stands out prominently, which was mentioned by the eminent English investigators of this type of alloys, and published by them in the 8th and 9th reports of the Alloys Research Committee of the Institution of Mechanical Engineers of Great Britain, is the fact that cast aluminum bronze possesses properties equal to those of rolled alu-

minum bronze. Nearly all copper-base alloys are improved by rolling processes, but the copper-aluminum alloys seem to possess equally good properties when cast or rolled; this is a remarkable metallurgical fact. Another important property of these copper-aluminum alloys is their resistance to alternating stress. Many tests indicate that their resistance is greater in this respect than that of some steels, and I have seen instances when cast aluminum-bronze bolts have outlived five steel bolts in foundation work subject to severe shocks. I mention these various instances to indicate that work originally started as research for substitution of one material for another frequently develops an article which has properties not possessed by the original metal or alloy.

I have dwelt particularly on the aluminum bronzes because recently I have done more special work on them than on other alloys, but I believe that the use of aluminum itself, in many combinations of metals, is a very important subject for investigation. Undoubtedly, after the war, the cost of aluminum will be reduced from its present price, and considering its low specific gravity it offers a very interesting and important field for research in the non-ferrous business. Naturally, if combinations containing aluminum can be developed in view of a probably increasing supply of metal, the cost will be reduced. This will benefit the industry generally and will immediately conserve tin.

#### Bronzes, Bearing Metals, and Solders

G. K. BURGESS\* and R. W. WOODWARD,† Washington, D. C.—From a metallurgical standpoint, there are several ways in which a reduction of the tin consumed in commercial non-ferrous and white-metal alloys can be effected. First, a reduction of the tin content of the alloy; second, substitution of part or all of the tin content by some other metal; third, a substitution of a different type of alloy, which in some cases also involves a change in mechanical design. The Bureau of Standards has been studying these methods of conservation for tin alloys, particularly in regard to babbitts and bearing metals, bronzes, and solders. Much of the information secured by the Bureau was obtained from answers to questionnaires sent to manufacturers and users of these materials, so that, in general, any of the following suggestions or recommendations can be considered as being practical and as having already been thoroughly tried.

#### *Bearing Metals*

There is no question that the tin content of nearly all bearing metals can be reduced to some extent, and in some cases actually eliminated

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† Assistant Physicist, U. S. Bureau of Standards.

without prejudice to the service rendered. The problem thus is to determine what needs are the most exacting, or when a breakdown would cause the greatest damage, and confine the use of high-tin babbitts to these uses. Thus the main bearings of airplane and military automobile engines, turbine shafts, etc., will probably have to continue to use high-tin babbitt, containing 84 to 91 per cent. tin. A babbitt metal such as S. A. E. specification No. 24, containing 84 tin, 9 antimony and 7 copper, appears to be as satisfactory in service as the genuine babbitt, 89 tin, 7.5 antimony, 3.5 copper, or that specified by the International Aircraft Standards Board, 91 tin, 4.5 antimony, 4.5 copper. But it should be pointed out that the latter two compositions are more fluid in the molten condition than the first named; consequently the lining can be made in a thinner shell with these babbitts, and the total amount of tin consumed may therefore be less than if the S. A. E. No. 24 were used. However, if the design of the bearing is not altered to admit of the thinner shell, the lower-composition babbitt should be used in general.

The following compositions (Table 1) are also recommended for use where a high grade of lining is required and where a genuine babbitt is now often used:

TABLE 1

	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
Tin.....	65	62	8	5	10 0	21 3
Antimony.....	..	..	8	7	12 5	....
Copper.....	3 to 6	4	4	2	0 5	3 0
Zinc.....	28 to 30	33	..	76	....	63.3
Aluminum.....	..	1	..	..	....	....
Lead.....	..	..	80	10	77.0	12 0

No. 3 and 4 have been found to do the service required of tin-base linings in machine-tool bearings; No. 5 can be used on similar bearings where a greater strain is met. No. 6 is in use in Germany as a "best" babbitt to conserve both tin and copper.

For linings on railroad-truck journals two compositions are in general use, one composed of 85 lead, 10 antimony, 5 tin, and the other of 87 lead, 13 antimony. The latter is restricted by some roads to freight service while the former is used on passenger equipment. Many roads, however, use the 87 lead, 13 antimony, on both classes of service and it seems that its use might be made more universal.

Another type of lining metal which deserves serious consideration is one composed almost entirely of lead, with small additions of alkali or alkali-earth metal. Certain of these have been given service tests at the Bureau of Standards and in many respects were found equal to or superior to genuine babbitt. Table 2 is a summary of the tests on such a

metal, and corresponding tests on genuine babbitt of composition 89 tin, 7.5 antimony, 3.5 copper:

TABLE 2

Load Lb. per Sq. In.	R.p.m.	Total Revolu- tions	Final Temp.		Rise in Temp.		Fric- tion, Lb.	Loss in Weight, Gram	Remarks
			°C.	°F.	°C.	°F.			
			Genuine Babbitt						Belt slipping. Bearing seized and smoking.
100	694	12,230	89	192	53	95	22	0.023	
200	706	16,510	102	216	58	104	29	0.021	
300	682	15,150	125	257	100	180	38	0.013	
400	603	6,600	139	282	94	169	79	0.054	
			Ulco Hard Metal						Bearing still good condition.
100	710	13,160	56	133	23	41	13	0.013	
200	715	18,870	69	156	33	59	18	0.021	
300	719	18,830	80	176	42	76	27	0.013	
400	711	17,310	81	178	43	77	23	0.022	
500	723	17,660	79	174	43	77	25	0.014	
600	692	14,960	84	183	45	81	24	0.021	
700	648	24,520	62	144	38	68	24	0.020	
800	365	12,870	53	127	20	36	23	0.010	
900	408	22,300	59	138	22	40	24	0.015	
1000	405	23,200	66	151	36	65	22	0.014	

*Standard Grades of Babbitt Metal*

At a meeting called by the Conservation Division of the War Industries Board on Apr. 15, 1918, which was attended by manufacturers and users of bearing metals, the Bureau of Standards was requested, after conference with technical representatives of the large manufacturers and users, to determine whether four classes of babbitt metal could be adopted, ranging in tin content as follows:

	PER CENT. TIN
A, Genuine Babbitt.....	89
B.....	40 to 50
C.....	4 to 6 5
D. ....	None

The Bureau has gone over the situation with several of the representatives, and the general opinion seems to be that it is impossible to limit some of the classes to a single composition, because of the fact that several compositions of nearly the same tin content are in general use for different purposes. Thus, in the table below, No. A-1 is used in aircraft engines, No. A-3 is used for automobile engines, No. A-4 is found in bearings of electrical machinery. It was thought, however,

that class B can be entirely dispensed with, as these intermediate-tin bearing metals are in no way as satisfactory as either a high-lead or a high-tin babbitt. In all cases where class B could be used, classes C or D will be found to serve the purpose equally well. There are, however, some grades of babbitt containing about 65 per cent. of tin which do not fall into either class A or class B, but are often claimed by the manufacturers to equal the high-tin babbitt in performance. If these claims can be substantiated, this babbitt should be considered as falling into the category of class A and as being a substitute for alloys in that class.

It should not be presumed, because high-tin babbitt of class A is included, that the Bureau recommends the continuance of its use for many bearings in which it is now used. The lowest possible tin alloy should always be used, and the Bureau believes that it might be advisable to allow some central body to issue licenses for the use of babbitt in class A, in order to insure that no A babbitt is being used where others are satisfactory.

Alloy D-2 has been included in class D because this comprises babbitt metals containing no tin. It should be noted that this alloy will be found satisfactory in many installations where class A has hitherto been used, and its inclusion in class D should not give the impression that it is a low-grade babbitt.

The American Society for Testing Materials has drawn up specifications for 12 compositions of babbitt metals (B23-18T) which, however, do not take into consideration the factor of tin conservation, but are formed for use in peace times. The present recommendations are for use in the existing situation, where the saving of all tin possible is of prime importance.

The recommended compositions for the various classes are given in Table 3. These have been selected so as to include existing specifications and usage as far as possible. Much information has been secured from the questionnaire sent out by the Bureau and from replies to the general letter issued by the War Industries Board on May 29, 1918.

TABLE 3

Class	No.	Tin, Per Cent.	Antimony, Per Cent.	Lead, Per Cent.	Copper, Per Cent.	Iron, Per Cent.(a)	Arsenic, Per Cent.(a)
A	A-1	91	4.5	1(a)	4.5	0.08	0.10
	A-2	89	7.5	1(a)	3.5	0.08	0.10
	A-3	84	9.0	1(a)	7.0	0.08	0.10
	A-4	83	8.5	1(a)	8.5	0.08	0.10
C	C-1	5	10.0	85	0.5(a)	....	0.20
D	D-1	..	13.0	87	0.5(a)	....	0.25
	D-2	(b)	(b)	98(b)	(b)	(b)	(b)

(a) Maximum.

(b) Remainder is alkali and alkali-earth metal.

More than traces of impurities other than those listed above will not be allowed, the following variations above or below the specified amount will be permissible for the desired elements:

PERCENTAGE OF ELEMENTS SPECIFIED	PERMISSIBLE VARIATIONS FROM SPECIFIED VALUE
Not over 5 per cent.	0.50
5 to 10 per cent., incl.	0.75
Over 10 per cent.	1.00

Large quantities of phosphor-bronze of the composition 80 copper, 10 lead, 10 tin, deoxidized with phosphorus, are used in unlined bearings at fairly high speeds and pressures. The following compositions (Table 4) have been suggested as substitutes for this composition, although it is our opinion that trouble will sometimes be experienced with Nos. 8, 9, and 10, because of their high lead, and these have about the same tin content as the others in the list.

TABLE 4.

	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12
Copper.....	81	79	74	64	Remainder	Remainder
Tin.....	7	5	5	5	8	5.0
Lead.....	9	15	20	25	15	17.5
Zinc.....	3	..	..	..	1.5-3	5.0
Antimony.....	..	..	..	5	...	....
Phosphor-copper	..	1	1	1	...	....

### *Structural Bronzes*

Considering bronzes other than those used for bearing purposes, we find that "Government Bronze" (Navy Specification, 46M6a, Gun Metal), or 88 copper, 10 tin, 2 zinc, is used in large quantities and can be modified to admit of a saving of tin without impairment of the physical properties sought. Experiments by the Bureau of Standards and others have shown that a composition of 88 copper, 8 tin, 4 zinc, is equal or superior to the ordinary Government bronze. Aluminum bronze, of composition 90 aluminum, 10 copper, for example, can also be substituted for many uses of Government bronze; so also can manganese bronze and naval brass. Several aluminum bronzes containing small amounts of iron have also been introduced, which can be either cast or wrought and are now being employed by several former users of Government bronze.

Some manufacturers have raised an objection to the use of aluminum bronze because the scrap accumulating from this alloy, if it should become mixed with other metals, particularly valuable metals, would have a deleterious effect upon them. This is simply a problem in works



management, requiring proper sorting and routing of scrap. We know of several large manufacturers who make aluminum bronze castings in proximity to steam metal castings, who, by taking the proper precautions, have encountered no difficulties.

Table 5 gives some of the properties of the above mentioned alloys:

TABLE 5.

Alloy	Tensile Strength, Lb. per Sq. In.	Elastic Limit, Lb. Per Sq. In.	Elongation in 2 In., Per Cent.	Authority
Government bronze, 88 Cu, 10 Sn, 2 Zn.	38,860	12,250	25.2	Average of 30 tensile specimens poured in 5 different foundries. Tensile tests made at the Bureau of Standards.
88 Cu, 8 Sn, 4 Zn.....	39,220	11,000	30.6	Same as above; 26 specimens only.
Aluminum bronze.....	71,000	25,000	21.0	Corse & Comstock <i>Trans. Soc. Autom. Eng.</i> (1916) 11, Pt. II, 272-73.
Manganese bronze, U. S. N.	70,000	35,000	30.0	Navy Department Specification 46B15.
Naval brass.....	54,000 over 1 in. 60,000 below ½ in.	25,000  27,000	40 0  35 0	Navy Department Specification 46B6b.
Aluminum bronze with iron <sup>1</sup> (Sillman bronze)				
Wrought.....	84,400	14,000	11.5	Bureau of Standards.
Cast.....	78,850	11,500	14.5	Bureau of Standards.

<sup>1</sup> Cu, 86.4; Al, 9.7; Fe, 3.9.

At the suggestion of the Imperial Electric Co., Akron, Ohio, the Bureau also made tests on Government bronze in which half of the tin was replaced by an equal amount of nickel. The averages of several tests on these alloys are shown in Table 6.

TABLE 6.

Partial Composition	Ultimate Strength, Lb. per Sq. In.	Yield Point, Lb. per Sq. In.	Elongation in 2 In., Per Cent.	Reduction in Area, Per Cent.	Modulus of Elasticity, Lb. per Sq. In.
Cu 88, Sn 5, Ni 5, Zn 2.....	40,680	13,050	31.8	28.0	17,300,000
Cu 89, Sn 4, Ni 4, Zn 3.....	39,675	11,500	31.2	31.2	14,900,000

It will be observed that the above values are exceedingly good for this class of material, and that either of the above alloys can be used as a means of conserving tin.

Many small machine-part castings are made of bronze which can just as readily, or even sometimes better, be manufactured of brass, *i.e.*, copper-zinc alloys. Tin is also looked upon in many brass foundries as a cure-all for poor castings, and is often added to the mixture when trouble is encountered. This practice is not only questionable but should not occur in brass foundries; the cause of the poor castings should be determined and the proper remedies applied in a regular manner.

### *Solders*

The composition of a solder will vary with the use for which it is intended. Formerly, every mechanic believed that nothing was as good as "half-and-half," 50 tin, 50 lead. It is very seldom that a 50-50 solder is necessary in the present emergency, and its use should be eliminated. No solder over 45-55 should be used for hand soldering with the iron, and in the majority of cases 40-60 will serve the purpose. Most plumbers use 40-60 for making wiped joints, whereas 37.5- 62.5 is just as satisfactory for all such purposes. Up to 1.5 per cent. of the tin in a wiping solder can be replaced by antimony, although this element is objectionable in solder for other purposes.

In the manufacture of automobile or airplane radiators, very little solder of higher tin content than 40 per cent. need be used, and in many cases 35-65 solder is being used with success. For the canning industry, both in the manufacture and in the sealing of the can, 37.5-62.5 solder can be used with satisfactory results. Articles which are tinned previous to soldering can be tinned in a bath composed of the eutectic of tin and lead—63 tin, 37 lead. This composition will be found to be fluid and will not segregate as will certain other tin-lead baths.

### *Cadmium as Tin Substitute*

Cadmium appears a promising substitute for part of the tin in solders. The Bureau has been developing such a solder and laboratory tests, together with manufacturing experience, so far point to a composition of 80 lead, 10 tin, 10 cadmium as being practical for many of the purposes for which solder is required. This solder has been tried in the manufacture of tin cans, on roofing materials, and for electrical joints, with encouraging results in all cases. Before using it for food containers, however, it will be necessary to ascertain its toxic properties under various conditions. A test has also been made of it in the manufacture of automobile radiators, with most satisfactory results.

The tensile strength of the cadmium solder is about the same as that of 40-60 solder, but the ductility is approximately twice that of the ordinary solders. The point of complete liquation is only slightly higher than that of the ordinary solders, while the range of solidification is considerably greater. Table 7 gives some of the provisional data on these solders, as compared with tin-lead solders; the tensile properties are the average of four determinations made on a Scott testing machine, the rate of separation being about 12 in. per minute.

TABLE 7.—*Physical Properties of Cadmium-lead and Tin-lead Solders*

Composition	Initial Solidification, °C.	Secondary Solidification, °C.	Final Solidification, °C.	Specific Gravity	Equivalent Volume to 1 Volume, 50-50 Solder	Tensile Strength, Lb. per Sq. In.	Elongation in 2 in., Per Cent.
50 Sn, 50 Pb.....	210*	181*	149*	8.81	1.00	5698	20.3
40 Sn, 60 Pb.....	238*	181*	149*	9.47	1.07	5820	26.0
37.5 Sn, 62.5 Pb.....	245*	181*	149*	9.54	1.08	5383	28.8
90 Pb, 10 Cd.....	267†	....	249†	11.09	1.26	5000	37.5
80 Pb, 10 Cd, 10 Sn....	254	183	143	10.35	1.17	5727	52.3
85 Pb, 10 Cd, 5 Sn.....	257	202	141	10.67	1.21		
75 Pb, 10 Cd, 15 Sn....	....	....	....	10.26	1.16	5880	41.7

\* Rosenhain and Tucker, *Phil. Trans.*, Royal Society of London (1918) A 209, 89.

† A. W. Kapp: Ueber vollständige Gefrierpunktscurven binärer Metalllegierungen *Drude's Ann. der Phys.* (1901) 6, 754. .

Because of the preponderance of lead in the cadmium solder, its price is very reasonable; with the present market prices of the metals involved, it is thought that the 80-10-10 solder can be sold, at a profit at 35 c. per pound. It is also thought that plenty of cadmium can be produced as soon as the market for it is created, as American sources of cadmium are undoubtedly available which are not at present exploited.<sup>4</sup>

In meetings with manufacturers of materials containing tin, it is always brought out that the Government is the worst offender, and that many Government specifications call for a lavish use of tin, which is sometimes detrimental to the quality of the material manufactured. We believe that many of these specifications are being revised in order to conserve tin, but there is room for further improvement. As a means of reducing the consumption of tin by the Government, we would suggest the advisability of creating a joint committee of technical representatives of the various departments to pass upon or revise all Government specifications containing tin. Such a committee could be in close coöperation with the manufacturers, and would offer a better opportu-

<sup>4</sup> C. E. Siebenthal: Cadmium in 1917. *Mineral Resources of the U. S.* (1917) Pt. I, 49-53. Also "Sources of Cadmium in the United States," this Volume.

nity for the manufacturers to criticise the tin content of Government specifications than is now afforded in any of the Departments.

## DISCUSSION

G. H. CLAMER.—Dr. Burgess referred to the objection on the part of foundrymen to the use of aluminum bronze as a substitute for tin bronze, because the aluminum so introduced finds its way into the scrap pile and is the cause of a great many bad heats of metal. A foundry in Philadelphia, which keeps a very careful record of all bad heats, found that 90 per cent., at least, of bad heats are due to aluminum contents. The foundry makes principally copper, tin, and lead alloys, also some red-brass valve metal, etc. The presence of a very small amount of aluminum in mixtures of that kind makes them absolutely worthless, unless they go through a refining process to eliminate the aluminum, which of course is expensive. For years, methods of detecting the source of aluminum in mixtures has been sought. The scrap pile might contain anything from bird cages to valves and miscellaneous pieces weighing from a small fraction of an ounce to maybe thousands of pounds; the fact that such a very small amount of aluminum is injurious means that probably one casting in the whole charge of metal, which may be from several hundred pounds to a ton, is sufficient to cause the rejection of that particular heat; the difficulty is increasing every day, for more and more aluminum is being used in brass and bronze mixtures. Ordinarily it takes some years for brass to get back to the scrap pile, so that while probably 10 years ago we had only a comparatively few bad heats from that source, today the number is gradually increasing as the percentage of aluminum-carrying alloys is increasing.

R. T. ROBERTS,\* Elizabeth, N. J.—The wrought-metal manufacturers also are having trouble keeping aluminum-bronze scrap from being mixed with their brass scrap. The two alloys used in wrought-metal are 95 copper, 5 aluminum and 92 copper, 8 aluminum, which have practically the same color as the ordinary 2-1 sheet brass. It is absolutely impossible to run those alloys in an ordinary brass mill and not get them confused with the regular brass. Each alloy is greatly superior to phosphor-bronze, either in a hard or a soft condition. An ordinary 5 per cent. aluminum-bronze wire drawn down to 0.025 in. (0.63 mm.) will have a tensile strength of over 150,000 lb. per square inch (11,250 kg. per sq. cm.). The same is true of an aluminum-bronze sheet for spring purposes. The only way the Government can successfully have aluminum-bronze substituted for phosphor-bronze will be to select one mill to handle the whole output and do nothing else; this would make an immense saving of tin in this present crisis.

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\* Waclark Wire Co.

## Babbitts and Solder

G. W. THOMPSON,\* Brooklyn, N. Y.—This subject has two aspects, neither of which can be ignored: these are the economic aspect and the technical aspect. Under ordinary conditions, economic law will take care of the conservation of tin. Under present conditions it seems desirable that economic law should still be permitted to operate as far as is practicable. It is true that this law operates rather slowly, and that under war conditions it cannot be depended upon to give our Government the supply it immediately needs. There should, therefore, be a designation of the essential industries by the Government, and tin should be supplied for those needs, letting the non-essential industries take what is left. At the same time, information ought to be forthcoming as to how tin can be conserved; with tin selling at 80 c. per pound, more or less, it is to the interest of every consumer to get the best information he can as to how he can save tin. Economic law would properly punish those who do not study their own interests in this way. I deprecate, therefore, any centralized socialistic effort toward avoiding penalties through failure to observe and obey economic law. In saying this, however, I am not unmindful that it is desirable for our Government to take such control of the tin situation as may be necessary to the prosecution of the war, practically regardless of the effect of such action upon individual industry, which should studiously seek to adjust itself to the new condition. The Government should promulgate such technical information as it can collect, showing how tin can be conserved, and should urge upon every consumer the exercise of his common sense in self-protection. There is very little danger of any one attempting to hoard or corner tin, in the present state of the market. Most consumers will be glad to live from hand to mouth, covering their sales by purchases, or *vice versa*.

As to the technical aspects: Tin and tin alloys are used to give certain practical and also certain artistic results. The practical factors are those involved in proper adhesion, continuity of surface, protection, the right degree of hardness, proper working qualities, etc. The artistic results are those that appeal purely to the eye or to our cultivated sense of what is desirable. There is no doubt that a great deal of tin could be saved if there were not a demand for certain pleasing effects. How can this saving be brought about? The users of tin bearing alloys are not the only ones involved in this question. The manufacturers of solder and babbitt have for a long time sought to give certain appearances to their fabricated bars and ingots in order to make them more salable. The user of a solder is very apt to judge of its working qualities by the appearance of the bar; the same thing is true with regard to babbitts. Under ordinary conditions, manufacturers are justified in trying to

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\* National Lead Co.

produce attractive and consequently more salable products. It would seem, however, that if solder and babbitt were cast in closed molds, just as good practical results would be obtained by the user, without his being able to give preference to solders of unnecessarily higher grade on account of their appearance. It would be hopeless for any one manufacturer of solder to undertake a change of this kind, but if all manufacturers of solder agreed upon it, they would be able, in my opinion, to get the users of solder to take, and to approve by their practical tests, metals containing less tin than they have been accustomed to. The same is true with regard to babbitt metals.

An illustration of how, in the use of an alloy, appearance sometimes is deceptive, is to be found in the case of what may be called intermediate grades of babbitt. I am thoroughly convinced that high-tin or high-lead babbitts are better than those containing both lead and tin with relatively high percentages of each. A high-tin babbitt should not contain more than about 10 per cent. of lead, and a high-lead babbitt should not contain more than 10 per cent. of tin, unless the percentage of antimony can be correspondingly increased. The intermediate babbitts, such as those that contain from 30 to 70 per cent. of lead or tin, may give nice appearing castings that flow easily, but they have not the serviceability of the high-lead or high-tin babbitts. Their hardness diminishes very rapidly as the temperature rises, and they have a relatively low softening point due to the eutectic components present.

#### The Cadmium Supply of the United States\*

C. E. SIEBENTHAL,† Washington, D. C.—From being one of the most maligned of metals—a veritable bugaboo—cadmium has almost overnight become respectable, though its slender claim to respectability rests almost wholly on the possibility of its substitution for tin. Preliminary to any campaign for such substitution, particularly for enforced substitution, the possible supply of cadmium should be determined as closely as possible; for that reason the statistical inquiry of which this paper embodies the results was undertaken.

#### *Production*

Cadmium is marketed in two forms, as metallic cadmium, in sticks or bars, and as cadmium sulfide, the pigment. The metal has found its greatest field of use in this country as a component of an easily fusible alloy that is used in automatic fire extinguishers. The sulfide is used to some extent in paints but chiefly to give color and luster to glass and porcelain.

The metal was first made in this country by the Grasselli Chemical

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\* Published by permission of the Director, U. S. Geological Survey.

† Geologist, U. S. Geological Survey.

Co. in 1907. Since 1910, the production has grown rapidly until, in 1916, the output was over 135,000 lb. and in 1917 over 207,000 lb. The output of cadmium sulfide for the same years increased from 22,000 to 50,000 lb. The market for cadmium was a little slow in the first half of 1918, stocks accumulated to some extent, and production was somewhat curtailed. In the first eight months, the output was 118,700 lb. of metallic cadmium and 36,500 lb. of the sulfide. The stocks on hand Aug. 31 were 161,000 lb. of metallic cadmium and 23,500 lb. of the sulfide. Other companies now manufacturing one or both forms of cadmium are the American Smelting & Refining Co., the U. S. Smelting, Refining & Mining Co. (including the lead smelter at Midvale, Utah and the electrolytic zinc plant at Kennett, Cal.); the Krebs Pigment & Chemical Co.; and the Midland Chemical Co. To these will shortly be added the Anaconda Copper Mining Co., the Judge Mining & Smelting Co., and the Consolidated Mining & Smelting Co. (Ltd.), of Trail, B. C., with the possibility of still others. Several plants producing cadmium-bearing residues have sold them to other companies already equipped to recover the metal. This points the way to custom cadmium-reduction plants large enough to handle economically the product of those plants whose output is too small to justify individual reduction plants.

### *Sources*

There are several cadmium minerals, but none of these occur in profitable quantities as ores. The cadmium of commerce is derived from zinc minerals and ores, in almost all of which it occurs in minute quantity, the ratio being about 1 of cadmium to 200 of zinc. Cadmium behaves metallurgically almost the same as zinc and hence constitutes a fraction of 1 per cent. of almost all spelter. The sources of cadmium that have been utilized are zinc ores treated by fractional distillation, lead-furnace bag-house "fumes," and residues from the purification vats of electrolytic zinc plants and lithopone plants.

*Fractional Distillation of Zinc Ores.*—Prior to the beginning of production in the United States, cadmium was made principally by Germany, where it was derived from zinc ores by the method of fractional distillation, which has never been practised to any extent in the United States. As cadmium has a higher volatility than zinc, the first vapor to distil over, which is ordinarily caught as "blue powder," contains a greater proportion of cadmium than that going over later. This enriched "blue powder" by special treatment has yielded most if not all of Germany's output of cadmium. Aside from the United States, Germany is the only important producer of cadmium, and her annual output in the years just before the war was about 80,000 pounds.

In the early years of the war, spelter containing more than 0.05 per cent. of cadmium was supposed to be unsuitable for cartridge brass.

Most spelter distilled in the United States and elsewhere averages from 0.2 to 0.3 per cent. of cadmium, the cadmium content ranging from a trace to about 0.75 per cent. The distillers of cadmium-free ores and the electrolytic zinc plants were not able to produce all the high-grade spelter needed for munitions. In this exigency several processes were proposed or revived to remove the cadmium from zinc ores. In one process it was proposed to add carbon to the zinc concentrates near the end of their journey through the long roasting hearth and at the same time bring the temperature up to such a point that the cadmium would distil freely, but not high enough to drive off much zinc. The vapor coming off could be collected and further refined to recover the cadmium. Such a process offers a possibility of recovering some part of the 1500 tons of cadmium contained in the zinc sulfide concentrates produced in the Joplin district in 1917. The acceptance of spelter with 0.5 per cent. of cadmium as Army "high grade," however, took away the incentive to remove the cadmium, though this process was for a time put into practice at one smelter.

*Lead-furnace Fumes.*—All lead ores contain a certain amount of zinc. In but one important lead-producing district in the United States, the disseminated lead district of southeastern Missouri, is the quantity of zinc too small to pay to separate in ore-dressing. When lead ores are smelted, the infinitesimal quantity of cadmium in the charge is separated from the associated zinc, the latter going into the slag and the cadmium going over with the fumes and being caught in the bag house, together with the lead and arsenic which were in the fumes. When these fumes have accumulated in sufficient quantity they are ignited and burnt to a clinker. This is charged back into the blast furnace, and the cadmium returns over the same route, accompanied by the cadmium content of the fresh charge of ore. Thus the cadmium and arsenic are constantly enriched while the lead remains constant. At one lead smelter, which has been in operation 6 years, the bag-house dust carries 8 per cent. of cadmium. The percentage of cadmium in the bag-house fumes at different smelters ranges from less than 0.1 to 10 or 15 per cent. The rate of enrichment of course depends on the nature of the ore that is being smelted. When the fumes are sufficiently rich in cadmium and arsenic, the arsenic is removed in the arsenic furnace and the residues, which may contain as high as 30 or 40 per cent. of cadmium, are treated for the recovery of that metal. Not all lead smelters are equipped with bag houses, and some lead ores contain very little cadmium. Nevertheless the bag-house fumes from lead blast furnaces must in time become enriched to the point where cadmium can be recovered from them. Such fumes have been hitherto the main source of the cadmium produced in this country.

*Electrolytic Zinc-plant Residues.*—In the reduction of zinc ores by



leaching and electrolytic deposition of the zinc, the zinc solution, prior to electrolysis, is cleared of cadmium by precipitation with zinc dust, for the presence of more than 0.05 per cent. of cadmium in spelter prevents it being classed as grade A in the Navy specifications. As between a zinc plant and a lead plant treating the same quantity of concentrates the zinc plant would handle from four to ten times as much cadmium because of the greater amount of zinc involved. It can be seen, therefore, that electrolytic zinc-plant residues will soon be a very important source of cadmium. About one-third of the supply in sight at present will come from the zinc plants. As both the zinc and the cadmium are recovered electrolytically the question of metallurgic supervision is simplified.

Since the early part of 1915, the demand for high-grade spelter has been so great that a considerable quantity of prime western spelter has been refined to the high grade either by redistillation or by electrolysis. In electrolysis the anode muds contain the impurities of the original spelter anode, which consist of cadmium, lead, and iron. Some cadmium has been recovered from such muds.

*Lithopone-plant Residues.*—In making lithopone the necessary zinc is supplied by dissolving in sulfuric acid zinc ashes and galvanizer's drosses, refuse zinc oxide, or roasted zinc concentrates. The zinc sulfate solution thus obtained must be purified by precipitating out the deleterious ingredients, among which is cadmium. The cadmium residues are concentrated to a usable degree of richness and worked up or sold. The cadmium at lithopone plants is most conveniently recovered in the form of the sulfide.

*Brass-shop Fumes.*—A competent authority estimates that 3 per cent. of the zinc used in brass manufacture escapes in the form of fumes. One-half of this zinc escapes up the flues in the melting and may be precipitated by Cottrell apparatus or otherwise, but the other half escapes into the air in pouring from the melting pots and is lost. The Bureau of Mines estimated several years ago that 400 lb. of cadmium was lost daily in such fumes at Waterbury, Conn. It is likely that the quantity is less now, relatively at least, because of the prevailing wider use in brass-making of spelter practically free of cadmium.

The fumes are richer in cadmium than the spelter used, because of the higher volatility of cadmium. They average about 0.2 per cent. of lead, 2 per cent. of copper, 30 per cent. of zinc, and from 1 to 1.5 per cent. of cadmium. If such fumes were used in the manufacture of lithopone, the resulting residues would be much richer in cadmium than those obtained when zinc ashes are used.

### *Resources*

A canvass was made of lead smelters, electrolytic zinc reduction and refining plants, lithopone plants, and a few typical brass works, with the

object of ascertaining the yearly recovery of cadmium in fumes and residues, the accumulated stocks of such materials, and the maximum yearly capacity of the cadmium-reduction plants. Completeness cannot be claimed for the results, but fairly detailed replies were received from the larger producers. Little attention has been paid to the cadmium content of fumes and residues, especially at plants where it accumulates in small quantity.

According to the data in hand, cadmium is accumulating at lead smelters at the rate of 350 to 400 tons annually and the stocks of fumes contain over 750 tons of cadmium. At electrolytic zinc plants about 200 tons is accumulating annually and the stocks of residues contain about 400 tons. At lithopone plants probably 50 tons is produced yearly and about 25 tons is contained in stocks. In round numbers, then, 600 tons accumulates annually, and there is on hand approximately 1200 tons in stocks of fumes and residues. Not all the stocks are rich enough for the cadmium to be commercially recovered but perhaps material that carries 1000 tons is suitable for treatment. The average recovery of cadmium is about 75 per cent. We may therefore estimate 750 tons of recoverable cadmium in residues in stock. An increase in the number and capacity of electrolytic zinc plants will correspondingly increase the annual accumulation of cadmium.

### *Reduction Capacity*

The maximum capacity for metallic cadmium reported by producers is 29,000 lb. a month, or about 175 tons a year. This capacity will be increased by the entrance into the producing list of the electrolytic zinc plants mentioned above. If the price of cadmium and the demand for it should justify expansion, the producing capacity could no doubt be brought up with reasonable promptness to 500 tons or more yearly. The price of cadmium will be the deciding factor also in determining what grade of cadmium fumes can be worked at a profit.

### *Prices*

In 1875, cadmium was quoted in the United States at \$3.20 a pound. In 1886, the average price for the total output of Germany was 80 cents a pound; but in 1890 and 1892, it fell off to 38 cents. In 1897, because of certain purchases by the Imperial Government, the price rose to \$1.23 a pound. In 1907, when the United States began making cadmium, the German average price was 84 cents. The price in the United States went down to 53 cents in 1909, but it has been steadily rising since. In 1916, the average price was \$1.56 a pound; and in 1917, it was \$1.47.

The cost of material for tin and cadmium solders of various formulas is shown in the following Table 1.

TABLE 1.—*Cost of Material in 100 Pounds of Solder, Oct. 1, 1918*

	50 Parts Lead 50 Parts Tin Dollars	60 Parts Lead 40 Parts Tin Dollars	80 Parts Lead 10 Parts Tin 10 Parts Cadmium Dollars	92 Parts Lead 8 Parts Cadmium Dollars
Lead at 8.05 cents..	4.03	4.83	6.44	7.41
Tin at 80 cents.....	40.00	32.00	8.00	
Cadmium at \$1.50..	.....	.....	15.00	12.00
Total cost.....	44.03	36.83	29.44	19.41

The solder containing 80 parts lead, 10 parts tin, and 10 parts cadmium can be made as cheaply as the half-and-half solder with cadmium at \$3.00 a pound and as cheaply as solder containing 60 parts lead and 40 parts tin with cadmium at \$2.25 a pound. The solder containing 92 parts lead and 8 parts cadmium can be made as cheaply as the lead-tin solders with cadmium at \$4.00 and \$3.25 a pound respectively.

## DISCUSSION

M. L. LISSBERGER,\* New York, N. Y.—We have heard a good deal about replacing the tin in solder by cadmium, but we have not heard anything about using cadmium in bronzes, brasses, and many other places where cadmium is already found with the zinc. If, however, only 160,000 lb. of cadmium are available and it is desirable to conserve tin, why not substitute cadmium for tin in bronze first? Those who have worked in cadmium think little of it in a solder. Cadmium solder may have its uses in electrical work, when it is made in the form of wire or ribbon, and is melted with a blowpipe; but when used for bath work, cadmium is soon oxidized and the bath becomes unworkable. As soon as an attempt is made to mix cadmium and lead, the cadmium is converted into an oxide. A tin-lead solder is an absolutely permanent mechanical mixture; to use cadmium solder until its permanency is known is dangerous. In addition, the effect of cadmium solder on food is not known; but it is known that solder containing 80 per cent. lead should not be used in any food container where there is any danger of the lead getting into the food.

There are means of conserving tin without danger. Bearing manufacturers could use all the cadmium that will be available for the next 3 or 4 years if a method for thus using it could be found, and thus save the tin now used in bearings for purposes where a dependable substitute has not yet been found. The use of phosphor-tin frequently permits the use of less tin in a mixture, because in many cases tin is used merely to smooth the mixture. But one of the chief places to

\* President, Marks Lissberger & Son, Inc.

conserve tin is in the plant. Care should be taken to prevent its going into oxides, drosses, and other waste and not to use scrap that contains tin in mixtures that do not require tin. Scrap containing tin should be employed only in mixtures in which tin must be used.

CHARLES W. HILL,\* East Pittsburgh, Pa.—The use of lead-cadmium solders is complicated by the ease with which cadmium oxidizes. The addition of tin or zinc to lead-cadmium mixtures appears to reduce this tendency toward oxidation, so that these solders may be readily used in iron soldering and if precautions against overheating are taken they may be used in pot soldering.

Zinc is only slightly soluble in lead-cadmium mixtures. Increasing percentages of cadmium increase the tendency to oxidation and raise the cost of the solder without increasing its strength. Most of our experiments have been made with a solder composed of lead 90.8 per cent., cadmium 7.8 per cent., and zinc 1.4 per cent. We have experienced no difficulty in preparing these solders using the ordinary precautions of solder mixing. They are stronger than the lead-cadmium-tin solders especially at temperatures above 25° C. At 100° C., they seem to be from 50 to 100 per cent. stronger than half-and-half solder and about 30 to 50 per cent. stronger than pure tin. The tests were made by pulling apart two copper cylinders that had been soldered end to end. This strength at high temperatures is of value in soldering apparatus that will become heated from one cause or another, such as articles which are to be japanned or are heated by steam. In this respect the lead-cadmium-zinc solders are much superior to lead-cadmium-tin solders.

The lead-cadmium-tin solders cannot be fluxed well without the use of a metallic chloride flux such as zinc chloride. They give good results on certain metals with organic fluxes, such as rosin or glycerine, but the soldered joint is stronger when zinc-chloride fluxes are used. We have soldered tin cans with zinc-cadmium-lead solders without the use of a flux. The matter of flux is of extreme importance in many cases, because of the corrosive action of excess flux left on the soldered article. This is true with copper.

It is quite probable that any danger from the use of these solders for tin cans used for foods would arise from the lead content and not the cadmium, which, contrary to the opinion expressed, is quite a stable element, except at high temperatures. It would obviously be dangerous to employ such solders in contact with foodstuffs without sufficient experimentation. The lead content of solders can be lowered and it is quite possible that some of the higher cadmium solders will not be injurious to health and will not be attacked by the contents of food cans.

In conclusion it may be said that lead-cadmium zinc solders are

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\* Research Laboratory, Westinghouse Elec. & Mfg. Co.

entirely suitable for certain uses and even possess advantages over present solders at high temperatures, but they are certainly not suitable for all uses. However, it is conceivable that their use in many places will effect a material conservation of tin. It will probably be wiser to use cadmium in solders where good results have been obtained than in brass where this element will be a source of possible trouble.

F. F. COLCORD,\* New York, N. Y.—The cadmium-lead solder has a higher conductivity than the lead-tin alloy, which ought to make it advantageous in the manufacture of electrical machinery. In the case of solder for tin-can work, one of the high officials of the American Can Co. said that he fully intended to give it a good trial, and I do not believe they are going to reach hasty conclusions and run the risk of poisoning the public.

#### Solder, Its Use and Abuse

MILTON L. LISSBERGER,† New York, N. Y.—Solder is a mechanical mixture of tin and lead, a fact which is susceptible of very simple demonstration. A bar of solder of a grade even as low as 30 per cent. tin and 70 per cent. lead, passed through a buffing machine, will show a surface practically identical with that of a bar of second-quality or reclaimed tin. The buffings, on chemical analysis, will prove to be almost pure lead.

According to the best practice, solder is made in the following manner. Virgin pig lead is first melted, and when it is thoroughly liquefied, virgin pig tin is added, together with a small amount of flux; the latter is for the purpose of bringing to the surface the so-called "liver," consisting of impurities that may have remained in either the lead or the tin as a result of incomplete refining. The combined material, when completely liquid, is thoroughly stirred for some hours, and is then cast into small pigs. Just before casting, and continuously during this operation, the molten metal yields dross, consisting largely of the oxides of lead and tin; this should be carefully skimmed off.

After the pigs have cooled, they are taken to a smaller kettle, remelted, and cast into the desired shape for use; or if wires, ribbons, etc., are to be made, the solder is cast into slugs suitable for extrusion and rolling. During this second operation, the skimming of dross should be even more carefully done than at first.

Hand mixing has proved to be the only reliable method for the production of the best quality of solder, irrespective of its percentages of lead and tin. The best quality of solder is not necessarily that which contains the highest percentage of tin, but rather is that composition which performs best on the required piece of work. In order to produce

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\* U. S. Metals Refining Co.

† President, Marks Lissberger & Son, Inc.

a thorough mechanical mixture, it is necessary to stir for a long period; experience has shown that to perform this operation satisfactorily takes from 5 to 6 hr., irrespective of the quantity of material being mixed, and also irrespective of the proportion of tin in the mixture, whether 60 per cent., or as low as 30 per cent.

Throughout the casting process, what occurs is that the lead solidifies in skeleton crystals until the remaining liquid has the eutectic composition, when it freezes at a constant temperature as a mechanical mixture of tin and lead containing some tin in solid solution. It is remarkable how many shapes these skeletons take. The seeming explanation of this variation is the presence of other metals than tin and lead, in very small proportion, or even traces.

An analysis, made in 1901, of borings taken from a section of a pig of solder at the points indicated in the accompanying diagram<sup>1</sup> showed the tin content to be as follows: No. 1, 59.06 per cent.; No. 2, 52.99 per cent.; No. 3, 38.43 per cent.; No. 4, 39.07 per cent.; No. 5, 45.62 per cent.; No. 6, 39.33 per cent.; No. 7, 38.82 per cent. Table 1 gives the breaking stress of twenty-five grades of wire solder of No. 9 Birmingham gage; tin wire of this gage breaks when subjected to a stress of 120 lb. and lead wire, when subjected to a stress of 49 lb. Table 2 gives the bursting pressures per square at different temperatures for twenty-four grades of solder; in each case the pressure is dead, not expansive.

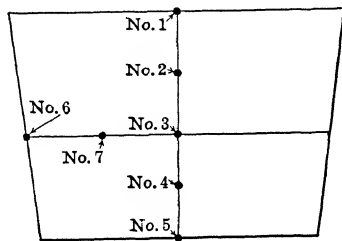


DIAGRAM SHOWING POSITION IN FIG OF BORINGS.

In arriving at these figures, it was impossible to use an expansive test because, after a number of experiments, it was observed the figures differed materially. It was then determined to use dead pressure, and this was found accurate with an atmospheric temperature of 56° F.; but when the tests were subjected to heat at 212° F. it was discovered that the length of time to which the test was subjected in this degree of heat made an important difference in the amount of pressure required to produce the burst. When subjected to 240° F. the difference was found still greater, and in both cases the longer the test was subjected to heat the less pressure was required to produce the fracture. The figures giving the bursting points at 212° and 240° must, therefore, be regarded as only approximately correct.

With the idea of conserving tin, solders should be separated into two classes:

<sup>1</sup> For this diagram and the tables appended we are indebted to Dr. G. W. Thompson of the National Lead Co.

TABLE 1.—*Breaking Stress of Wire Solder of No. 9 Birmingham Gage*<sup>2</sup>

Tin, Per Cent.	Lead, Per Cent.	Breaking Stress, Pounds	Tin, Per Cent.	Lead, Per Cent.	Breaking Stress, Pounds
25	75	65	48	52	93
28	72	68	50	50	95
30	70	69	52	48	97
33.3	66.6	73	54	46	98.5
35	65	74	56	44	101
36.5	64.5	78	58	42	103
37	63	79	60	40	105
38	62	80	62	38	106.5
38.5	61.5	78	64	36	108
40	60	84	67	33	109.5
42	58	85	70	30	112
44	56	87	75	25	115
46	54	91			

TABLE 2.—*Bursting Pressure per Square Inch of Solder at Different Temperatures*

Tin, Per Cent.	Lead, Per Cent.	At 56° F., Pounds	At 212° F., Pounds	At 240° F., Pounds
25	75	362	146	74
28	72	384	158	77
30	70	417	170	84
32	68	436	179	88
33.3	66.6	443	181	89
35	65	460	188	93
37	63	476	196	97
38	62	482	199	99
40	60	493	202	100
42	58	497	208	103
45	55	505	212	105
46	54	508	213	106
48	52	515	215	107
50	50	522	220	109
52	48	527	223	111
54	46	527	223	111
56	44	528	223	111
58	42	533	227	113
60	40	533	227	113
62	38	535	227	113
64	36	539	227	114
66	34	541	227	114
70	30	550	230	115
75	25	561	235	118

<sup>2</sup> Experiments made by F. W. Schultz and recorded in his book on Solder published in 1908.

(1) That which is used strictly for soldering, that is, joining and holding together two pieces of metal.

(2) That which is used primarily for the filling of an interlocked joint, so as to prevent the escape of the contents of a container. It is these filling metals that offer the greatest opportunity for the conservation of tin. It is only necessary that the metal shall flow into the seam, and solidify into an impenetrable mass.

The greatest abuse of solder occurs in the use of high-tin mixtures for filling metals. A mixture of 25 per cent. tin and 75 per cent. lead, worked at the right temperature and with proper fluxing, is high enough in tin for any filling purpose, as has been demonstrated in the practice of the oil canners, notably the Standard Oil Company.

The filling operation is usually conducted by machinery, but the users have frequently not realized that the baths are considerably richer in tin at the top, through which layer the container is being dragged, than the solder that is put into the baths. When the 40:60 solder, most commonly used on automatic can-making machinery, has not worked entirely satisfactorily, it has often been found that the addition of 1 or 2 in. to the depth of the bath has made the solder work very much better. Hence, one of the best means of conserving tin in can-making solder is to deepen all baths, whether on line machinery or for hand dipping, thus permitting the use of a lower-grade solder. Owing to the increasing adoption of the so-called "sanitary can" in the food-canning industry and of the interlocked seam for oil and other containers, it is safe to estimate that over 75 per cent. of the entire consumption of solder is used as filling; hence, the possible saving of tin in this direction becomes highly important.

The fact that solder dross contains a higher percentage of tin than the original solder has usually been explained on the assumption that tin oxidizes more rapidly than lead. The probable explanation is that in solder baths the lead is gradually working toward the bottom and the tin to the top, where it is exposed to the oxygen of the air; thus the oxide of solder is richer in tin than the original solder.

The overheating of solder is not only detrimental to the work, but also causes some, though not a very great, waste of tin through the excessive production of oxide. While this oxidation may be a source of considerable expense to the package manufacturer, it is not actually a very serious loss of tin because the reclaiming of these drosses, or oxides, has been so perfected that very little of the original metallic contents is lost. In these days, however, when every ounce of tin should be conserved, both to insure a sufficient supply for the most essential work, and to save the useless transportation of a material which comes such long distances by boat, overheating should be avoided, and all baths should be covered with a protecting material such as sal ammoniac, oil, charcoal, or ash.



In this connection, it should be emphasized that every particle of solder oxide should be preserved, and sent to the reclaimer. A teaspoonful of solder dross contains enough solder to make a 5-gal. can or 100 No. 1 cans. In many plants, even those of some of our largest consumers of solder, this dross is not collected and saved with sufficient care. It is seldom that a thorough cleaning and gathering together of the oxides takes place more than once a week.

Fire will purify these reclaimed materials, when properly refined, and in purity they will compare favorably with the virgin materials. However, too little attention has been paid to the proper refining of these scrap metals. Usually they have simply been put into a kettle, melted down, and then brought up or down to the required composition. This is not sufficient. Reclaimed metals are never equal to virgin metals, no matter how much refining they undergo; nevertheless, for certain classes of work they are economical and efficient. The repeated use of metal affects its physical permanency; yet the margin of safety in the use of solder is so large, and the length of time that solder is required to remain on the container is so comparatively short, that any lack of permanency can usually be safely disregarded.

After many years of experience, we have developed the following method of manufacture. The lead is first melted at a temperature which does not cause too rapid fusion. After the dross has had a chance to rise to the surface, it is carefully skimmed off before the required amount of tin is added, and slowly reduced to the liquid state. From the moment the tin is added, the solder is stirred by hand for 3 to 4 hr. A scavenger is then added and thoroughly worked for another 3 to 4 hr.; the resulting dross is again skimmed, and the solder is cast into pigs of approximately 80 lb. each. These pigs are then remelted in smaller kettles at a temperature which just causes free fluidity, and the solder is then cast into the desired shapes. During the entire operation of final casting, the caster stirs every time he takes a ladleful from the pot.

This work could be done much more rapidly at higher temperatures, and much more economically by the use of mechanical mixers, but the resulting solder would not be so thoroughly mixed, nor would it be so fluid. Mechanical mixing has a tendency to drive the oxide and dross back into the metals, thus diminishing the holding power of the solder. The scavenger must be chosen with great care, and the amount must be very accurately gaged; otherwise the scavenger becomes a constituent of the finished product, and, instead of being beneficial, is a detriment. We have found that in the grades of solder containing 46 per cent. and less of tin, the addition of  $\frac{1}{2}$  to  $\frac{3}{4}$  per cent. of the best grades of antimony increases the fluidity and holding strength of the solder for working tin plate.

Next to its use for containers, the largest consumption of solder

has been on gasolene motor radiators. The hand work on these radiators requires merely a free-flowing clean solder, but on the dipping work, where most of the solder is used, the greatest abuse has been practised. As these radiators are composed of copper, low brass, or ordinary brass, no antimony whatever should be added to the solder used for this purpose. Also the affinity of tin and lead for zinc and copper will draw both of these metals from the radiators into the baths, and as both copper and zinc make solder sluggish, it does not take long (unless proper methods are employed for cleansing the baths) for the solder to become deteriorated.

These baths can be thoroughly cleaned by a mixture of rosin and sulfur, but as this operation produces very disagreeable black smoke throughout the plant, some method should be devised for disposing of it. When sulfur is used for removing zinc and copper, a sufficiently high temperature should be employed to insure the complete combustion of the sulfur. The baths should then be allowed to settle for at least half an hour after such heating, and the top carefully skimmed to remove any sulfides present. It is important to note that the presence of any non-metallic substance is injurious to solder, whether it has been added as a scavenger or is liberated from the original metals.

The question is frequently asked, what is the strongest solder that can be made? Numerous experiments have been made, but the results are confusing. Tests of tensile strength, based upon wires and cast bars, indicate that the higher the tin, up to 75 per cent. tin, 25 per cent. lead, the greater the breaking strength; in the case of two pieces of tin plate soldered together, the maximum strength is given by a solder containing around 42 per cent. tin. Other tests were made on square 5-gal. cans, completely filled with water and then capped; when dropped from a height of about 100 ft., the cans soldered with 46 per cent. tin, 54 per cent. lead, in no case broke at the seams, although the tin plate was ruptured. This was the only mixture that gave this result. Cans soldered with 47 per cent. or more of tin, 53 per cent. or less of lead, and with 45 per cent. or less of tin, 55 per cent. or more of lead, occasionally ruptured at the seams. These experiments were made most carefully and were afterward confirmed by subjecting the cans to air pressure.

I am thus inclined to believe that, in round figures, 46 per cent. tin, 54 per cent. lead, is the strongest mixture that can be used for general soldering purposes, particularly if  $\frac{1}{4}$  to  $\frac{1}{2}$  per cent. of antimony be added to the mixture. The Bureau of Standards, with the approval of the War Industries Board, suggests that the highest grade of solder permitted should be 45 per cent. tin, 55 per cent. lead. For mechanical soldering, 40 per cent. should be the highest tin ratio, and for most bath work it has been demonstrated that tin from 35 per cent. to 38 per cent., according to the nature of the work, will give ample satisfaction, provided the solder is made properly.

### Constitution of Tin Bronzes

BY SAMUEL L. HOYT,\* E. M., PH. D., MINNEAPOLIS, MINN.

(Milwaukee Meeting, October, 1918)

THE writer has long been interested in seeking an explanation of the upper heat effect in the copper-tin alloys over the  $\alpha + \beta$  range, first described in 1913. These notes are offered, not at all as the final explanation of this heat effect, but rather to indicate certain progress which has been made toward establishing what happens over this temperature interval.

While working on the thermal analysis of the copper-rich kalchoids (copper-tin-zinc alloys), it was noted that those alloys containing major portions of tin and minor portions of zinc exhibited two marked heat effects, one at about 520° C. and the other at about 600° C., instead of the single effect which is generally observed in either of the two binary series. This matter was discussed with Dr. Guertler, who suggested that a heat effect at about 600° C. in the pure copper-tin alloys might be expected. This led to a more searching examination of the thermal critical points in the pure copper-tin alloys, with the result that a marked, although somewhat weak, heat effect was located at about 600°. Somewhat later, this upper heat effect was discussed with Dr. Burgess, and it was requested that the Bureau of Standards make heating and cooling curves of one of these alloys for the purpose of removing any possible doubt as to the actual presence of the heat effect. Results obtained by the Bureau are given in Plate I.

In an earlier paper<sup>1</sup> differential curves were published showing that the heat effect occurred at constant temperature over the  $\alpha + \beta$  range and increased in magnitude with the amount of  $\beta$  up to a point somewhat above 20 per cent. tin. No heat effect was noticed at this temperature in the eutectoid alloy.

The existence of this heat effect seems to require a modification of the present copper-tin diagram in the  $\alpha + \beta$  field, and it has been the object of the work here presented to secure the evidence upon which such a change should be based.

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\* Associate Professor of Metallography, University of Minnesota.

<sup>1</sup>S. L. Hoyt: On the Copper-rich Kalchoids. *Journal, Institute of Metals* (No. 2, 1913) 10, 235.

Inasmuch as this upper heat effect is much more marked in the ternary alloys, and is probably due to the same cause, a small bar, 4 in. (10.16 cm.) long, of an alloy 75 Cu, 15 Sn, 10 Zn, was heated over its entire length to a temperature of about 700° C., or well above the upper heat effect. The bar was then slowly moved along to the cooler portions of the furnace, and finally partially removed from the furnace so as to cause the temperature of one end to fall gradually from 700° C. to

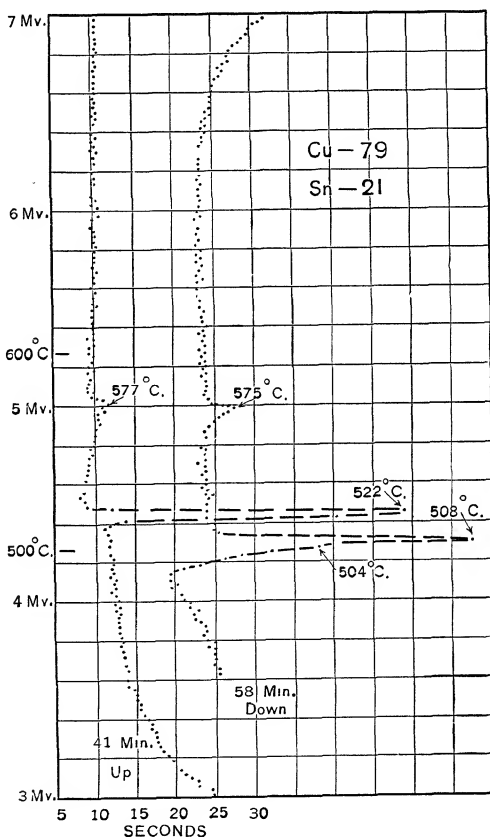


PLATE I.

about 400° C., while the temperature of the other end remained about as before. The bar was held in this condition for 8 hr. and then suddenly quenched in cold water. One side was ground down, polished, and etched with ferric chloride.

As was expected, the complete transition from the high- to the low-temperature modifications could be observed. These are reproduced in Figs. 1 to 6. Fig. 1 shows the high-temperature modifications, which may be called  $\alpha$  and  $\beta$ . At a position corresponding to a somewhat

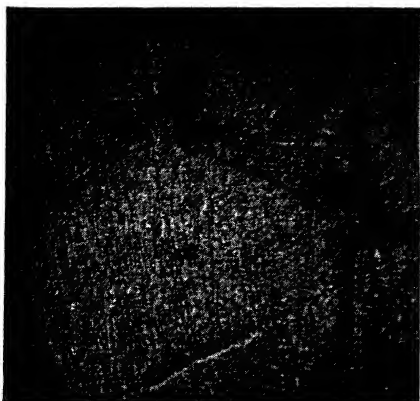


FIG. 1.—HOT END.

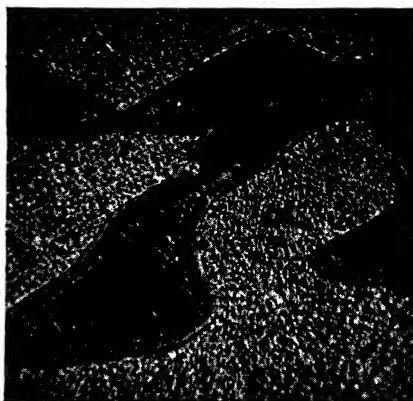


FIG. 2.

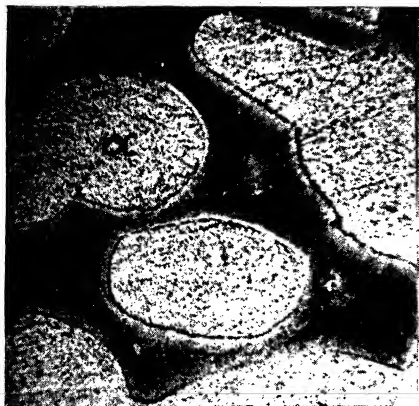


FIG. 3.



FIG. 4.

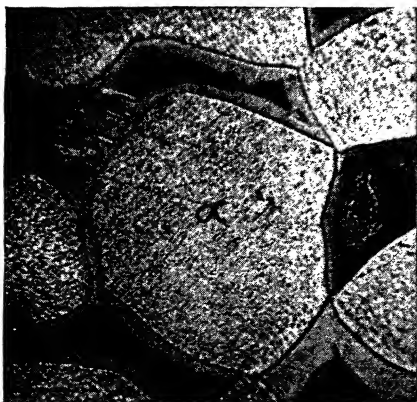


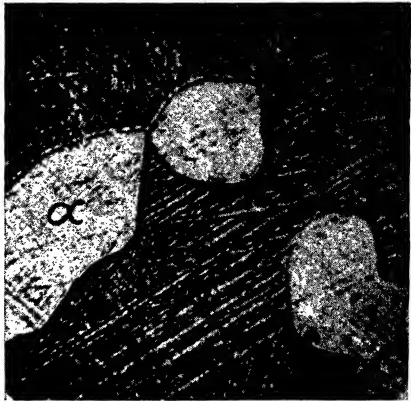
FIG. 5.



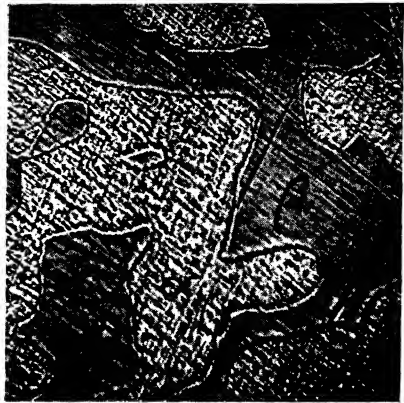
FIG. 6.—COOL END.

FIGS. 1-6.—ALLOY 75 CU, 15 SN, 10 ZN. ETCHED WITH FERRIC CHLORIDE.  $\times 800$ .

lower temperature, a well defined reaction rim is formed between  $\alpha$  and  $\beta$ . At a still lower temperature this reaction rim has grown somewhat, while smaller crystals of the new phase have formed at the central parts of the original  $\beta$  constituent. While this new phase has the appearance of being a reaction rim, it must be conceded that proof that it is such is yet lack-

FIG. 7.— $\times 100$ .FIG. 8.— $\times 800$ .

FIGS. 7-8.—ALLOY 79 CU, 21 SN. QUENCHED FROM ABOVE UPPER HEAT EFFECT. ETCHED WITH FERRIC CHLORIDE.

FIG. 9.—ETCHED WITH FERRIC CHLORIDE.  $\times 100$ .FIG. 10.—ETCHED WITH CUPRIC CHLORIDE.  $\times 800$ .

FIGS. 9-10.—ALLOY 79 CU, 21 SN. QUENCHED FROM BETWEEN THE UPPER AND LOWER CRITICAL POINTS.

ing. At a position still further toward the cold end, we find additional complication in the succession of phases. The reaction rim has encroached still further upon the original  $\beta$  constituent but, in turn, has given way to a fourth phase which, like the third, is light gray in color, the  $\alpha$  being yellow and the  $\beta$  being brown, on etching with ferric chloride. The four phases are shown very clearly in Fig. 4.

In Fig. 5, which shows a field still further toward the cold end, we find the beginning of the formation of the eutectoid structure, with the last traces of the original reaction rim, or possibly, in exceptional cases, of



FIG. 11.—ALLOY 75 CU, 25 SN. QUENCHED FROM ABOVE THE UPPER CRITICAL POINT. ETCHED WITH ROSENHAIN'S REAGENT.  $\times 75$ .

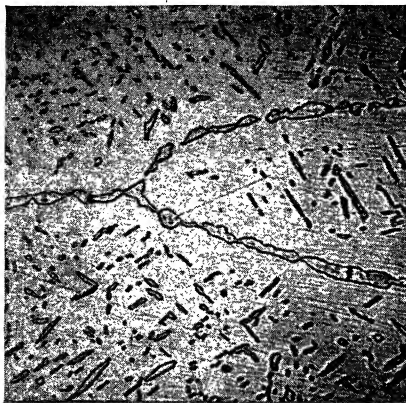


FIG. 12.—ALLOY 75 CU, 25 SN. QUENCHED FROM BETWEEN THE UPPER AND LOWER CRITICAL POINTS. ETCHED WITH CUPRIC CHLORIDE.  $\times 100$ .

the original  $\beta$ . Apparently, at this point, what was originally the reaction rim changes over into the eutectoid. At a point slightly nearer the cold end, as in Fig. 6, we find the normal structure of the slowly cooled



FIG. 13.—ALLOY 75 CU, 25 SN. QUENCHED FROM BETWEEN THE UPPER AND LOWER CRITICAL POINTS. ETCHED WITH CUPRIC CHLORIDE.  $\times 800$ .

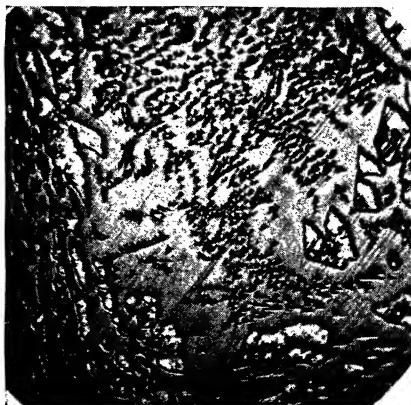


FIG. 14.—ALLOY 75 CU, 25 SN. ETCHED WITH FERRIC CHLORIDE.  $\times 800$ .

alloy, except that, instead of there being three generations of  $\alpha$ , there are now only two, presumably the first and the second.

It would do little or no good to attempt to analyze the structural

changes from the point of view of the phase rule, inasmuch as we are quite evidently not dealing with stable equilibria. As an explanation of the two well defined heat effects exhibited by this alloy, these photographs also offer little that is enlightening. Thus it seems apparent that the tin-rich constituent of the eutectoid may make its appearance prior to the actual formation of the eutectoid, which certainly is not in accordance with our ideas of eutectoid formation.

Two pure copper-tin alloys, one containing a considerable proportion of excess  $\alpha$  and the other only a slight amount, were quenched in water from above the upper critical point and from between the upper and lower critical points respectively. The results obtained are shown in Figs. 7 to 13. Figs. 7 and 8 show the customary  $\alpha$  and  $\beta$  phases. Figs. 9 and 10, except for the larger amount of  $\alpha$ , show the same structure, i.e.,  $\alpha + \beta$ . Fig. 10 shows the effect of etching with cupric chloride, which reverses the action of ferric chloride and attacks the  $\alpha$  instead of the  $\beta$ . A similar structure is obtained by preliminary heating to above the upper critical point prior to holding the temperature constant between the critical points.

In the course of this work, a fair idea of the conditions which lead to the formation of the second and third generations of  $\alpha$  has been obtained. These two generations, both found in eutectoid structures, were described in the paper already referred to, the alloy being a ternary alloy of copper, tin and zinc. The same occurrence in the binary copper-tin alloys is shown in Fig. 14.

## DISCUSSION

PAUL D. MERICA,\* Washington, D. C. (written discussion†).—Investigation that has for its purpose the determination of the constitution of the alloys of copper with tin and with zinc is not only of much scientific interest, but will serve a very practical purpose as well. The constituents, with which the anomalous heat effects discovered by the author are undoubtedly associated, are to be found in commercial brasses and bronzes, and can exert a profound effect upon their mechanical properties, although present in relatively small proportion.

Thus in a recent article<sup>1</sup> it was shown that a sample of commercial naval brass rod, which normally consists of  $\alpha$  and  $\beta$  grains, developed envelopes of the so-called  $\delta$  constituents around the  $\beta$  grains upon heating to 330° to 430° C. The effect of this new constituent upon the tensile properties of the rod was most striking; the elongation in 2 in. was reduced from about 35 per cent. to from 21 to 26 per cent.

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\* Metallurgist, U. S. Bureau of Standards.

† Received Oct. 8, 1918.

<sup>1</sup> P. D. Merica and L. W. Schad: Thermal Expansion of Alpha and of Beta Brass. *Journal, American Institute of Metals* (1917) 11, 396.



It is therefore apparent that in wrought brasses of similar composition, containing tin, there is the very unpleasant possibility of developing this  $\delta$  constituent by annealing, and thus spoiling the material. It becomes, thus, a matter of the utmost importance to determine the temperature limits of the appearance and disappearance of this constituent, and indeed to clear up the whole vexing question of its identity and relation to the constitution of the ternary alloy system.

A most significant feature of this  $\delta$  constituent is its development, possibly as a peritectoid at the edge of the  $\beta$  grains, forming a brittle envelope around them. It is undoubtedly a constituent to be avoided if possible, although I have tested wrought brass containing it in granular form, which was of most excellent mechanical properties.

I should like to ask the author whether he is aware of the existence of this constituent in commercial casting bronzes, such as the well known 88-10-2 bronze, and of its effect upon the mechanical properties of such bronze.

I am most interested in this line of work and shall hope that the author will not overlook the practical application of it.

C. H. BIERBAUM,\* Buffalo, N. Y.—I agree with Dr. Merica that the eutectoid has a distinct effect upon the alloy and also that, as yet, it is difficult to say at just what point this eutectoid occurs or the conditions under which it appears; that is, the percentages of copper, tin, zinc, lead, and the other elements together with the temperature of pouring and the rate of cooling that is necessary; all seem to have a contributing effect. Some authorities, Law for instance, gives it as 9 per cent. of tin, yet under certain conditions I have found it with a lower percentage of tin to copper contents than that. The hard delta and the soft alpha crystals are both necessary for a bearing alloy; the one supplements the other.

I am not inclined to think that phosphorus has any effect upon the eutectoid forming. The phosphide,  $\text{PCu}_3$ , forms in and around the delta but is and remains distinct and separated from it. Etching with ferric chloride and then with nitric acid shows this very distinctly; it darkens the eutectoid and the other parts and leaves the phosphide bright. The rate of cooling determines the size of the crystals and the eutectoid together with their distribution and orientation. It is possible to chill the alloy to such an extent where no eutectoid appears. I think it has been positively demonstrated that only an alpha and a beta appear when this copper-tin alloy has been poured between two polished ingots of copper and taking the exact point, as far as it would flow, almost a knife-edge, this very point after etching showed only an alpha and a beta as far as it showed anything; we found no sign of a delta. If the

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\* Lumen Bearing Co.

very edge or point of this alloy, cooled in this manner, is etched with either ferric chloride or with ammonia the surfaces show an entirely different structure. In the one case the high copper is obtained and in the other the high tin formation; in the one case there is a distinct crystalline formation, and in the other an almost amorphous condition. It becomes simply a question which reagent has been used for the etching.

The subject of bearings is ordinarily taken up from one point of view, that is, in considering the alloy only. The alloy used for a bearing should always be considered in conjunction with the corresponding bearing member, its composition and its hardness. If the steel journal is hard enough to receive the eutectoid crystal of the bronze, the bearing will be improved by it. On the other hand, if the steel is not hard enough the bearing will not be improved by the presence of the eutectoid.

S. L. HORT (author's reply to discussion\*).—The example cited, by Dr. Merica, of the importance, to the technical man, of the present investigation is extremely interesting and may lead others to pursue the work even further than the writer has been able to. As to the occurrence of the so-called  $\delta$  constituent in the bronze mentioned, Dr. Brinton and the writer have shown that the addition of zinc increases the magnitude of the upper heat effect so that, undoubtedly,  $\delta$  would be found in greater quantities in the ternary alloys than in the pure copper-tin alloys. Presumably its tendency to form the undesirable envelopes would likewise increase. A heat treatment to overcome the formation of  $\delta$  as envelopes, which might well be tried in technical practice, would be to quench from above the upper critical point and reheat to some lower temperature to produce the desired physical properties.

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\* Received Nov. 22, 1918.

## Electrolytic Zinc

BY C. A. HANSEN,\* SCHENECTADY, N. Y.

(Colorado Meeting, September, 1918)

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### INTRODUCTION

It has been the experience of the writer, during some five years' work with electrolytic zinc, that the zinc cell is perhaps more sensitive to impurities in the electrolyte than the analytical methods as yet developed for the chemical identification and determination of these impurities; but that, when proper purification methods have been applied to a given solution, the behavior of the cell is quite independent of the original sources from which the zinc sulfate has been derived.

It has also appeared that the behavior of the zinc cell is the best and surest check on the correctness and thoroughness of the purification methods used, because it indicates the presence of both current impurities and cumulative impurities derived from the leaching of the ore; it also indicates whether these impurities are current or cumulative, at least in so far as they are of serious importance commercially.

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\* Research Laboratory, General Electric Co.

It is the writer's opinion that, aside from the gradual reduction in the costs of producing power, it is the realization of the necessity for thorough purification of zinc solutions that has made electrolytic zinc commercially possible. Practically every detail of present zinc-plant practice has been applied to electrolytic zinc work during the latter half of the last century, and it is probable that mere lack of thoroughness in the manner of the application explains the comparative failure to obtain commercial results. It is also the writer's opinion that failure to realize the importance of thorough purification will be the primary cause of failure of zinc plants in the future.

In spite of a great deal of effort expended upon synthetic solutions, no inorganic addition agent has been found which improves the power characteristic of the zinc cell, nor any organic addition agent which improves a commercially pure solution except to a minor degree in that its use may result in slightly smoother deposits.

In view of this it appears important to learn definitely the characteristics of the zinc cell operating with commercially pure solutions; it also appears proper to make use of these cell characteristics as indicating the purity of a given solution.

The art of electrolytic zinc is still in the stage where the list of personal prejudices or guesses is considerably longer than the list of definitely established facts. For the reason that the graphic method of presenting observations exposes the extent to which personal prejudices have been permitted to swing the final conclusions from the observed facts, this method will be used so far as appears practicable.

### *Source of Materials Tested*

That the sources of the various solutions under discussion are widely diversified is indicated by the analyses of the ores and other products tested (Table 1). Gold and silver were present in them all.

## POWER CHARACTERISTICS IN ZINC SULFATE ELECTROLYSIS

### *Current Efficiency*

It has always appeared that there are but two major reactions which take place in the zinc sulfate cell using peroxidized lead anodes:

1. Deposition of zinc at the cathode at a rate which depends entirely upon the current density and varies according to Faraday's constant.
2. Corrosion of the deposited zinc by the cell solution, at a rate which is independent of the deposition rate, but which does depend entirely upon the chemical activity of the solution and upon the zinc surface exposed.

TABLE 1.—*Analyses of Ores, Etc., under Discussion*

	Bully Hill Ore	California Flue-dust	Australian Concentrate	Tasmanian Concentrate	Tasmanian Ore A	Tasmanian Ore B	Utah Concentrate
Zn.....	29.00	26.05	48.80	51.60	31.00	31.75	43.73
Fe.....	15.05	6.22	8.55	5.14	13.50	10.10	6.10
Cu.....	2.18	1.10	0.33	0.31	0.60	0.37	1.38
Cd.....	0.30	0.97	0.05	0.12	0.07	0.09	0.31
Pb.....	Trace	8.63	6.12	4.70	9.90	10.85	3.83
Sn.....	Trace						
S.....	31.10	7.70	27.30	30.00	34.40	26.40	30.40
SiO <sub>2</sub> .....	6.30	6.42	1.75	6.24	6.70	10.00	6.70
Al <sub>2</sub> O <sub>3</sub> .....	8.26	3.48	0.56	.....	2.00	2.20	5.22
BaSO <sub>4</sub> .....	2.92						
CaO.....	2.31	1.44	0.50	.....	1.20	0.40	4.95
MgO.....	2.86	0.36	0.06	.....	0.20	0.60	
MnO.....	0.60	0.04	1.03	0.60	0.40	0.80	2.50
As.....	0.00	7.35	0.04	0.08	0.37	0.15	0.52
Sb.....	0.00	1.13	0.08	0.03	0.09	0.12	0.16
Bi.....	.....	0.65					
Te.....	.....	1.06					
Se.....	.....	0.23					
Na.....	.....	0.26					
K.....	.....	0.34					
Cl.....	.....	0.06					
Co.....	.....	.....	Trace	0.00	0.00	0.00	0.00
Va.....	.....	.....	.....	.....	.....	.....	Trace

So far as concerns pure solutions, the nature of the zinc exposed is immaterial, being always considered a standard quantity. As the deposits become less smooth with increasing weight, they naturally do expose a greater actual surface to corrosion, and we should expect current efficiency to drop off with increasing length of deposition.

The corrosion rate is mainly determined by the acid concentration of the electrolyte. Fig. 1 shows the solution rate of pure zinc cathodes suspended in active cells, but insulated from the live busbars. It was assumed that the rate of solution in acid might be affected by the nascent oxygen given off at the anodes, and we were interested in the corrosion rate in solutions saturated with this oxygen rather than in what might take place in acid alone.

With a given acid concentration, it is also to be expected that the corrosion rate will increase with increasing temperature. This increase is, however, not so marked as to obtrude itself in the data at hand. It is probable that, like the corrosion of copper in acid sulfate solutions, and in acid ferric sulfate solutions (as shown by Addicks), the corrosion rate is about doubled with each 20° C. rise in temperature. If the above assumptions are correct, they should be reflected in the results obtained with different current densities. The effect of impurities in solution will be discussed later.

Table 2 summarizes typical results with solutions made from several different raw materials listed in Table 1, after suitable purification methods had been evolved for them. During all of the work detailed, five or more cells were operated in solution cascade and in electrical series, thus giving a set of interrelated data by each experiment. This interrelation

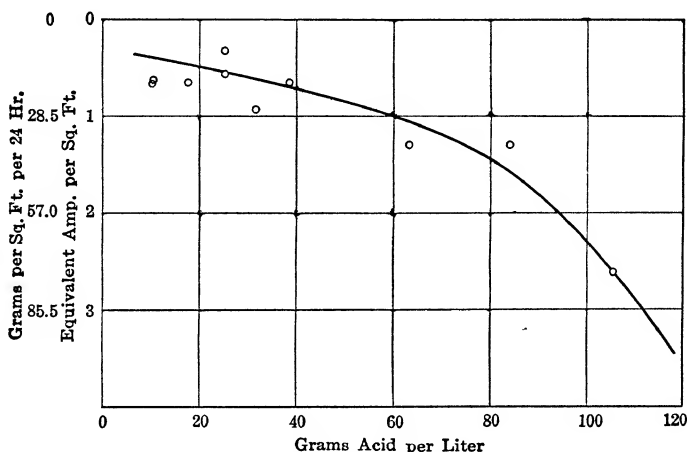


FIG. 1.—CORROSION OF SMOOTH CATHODES, SUSPENDED IN ACTIVE CELLS, BUT INSULATED FROM BUS BARS. BULLY HILL, MARCH, 1915. (C. A. Hansen.)

has proved to be a most excellent check on the individual cell results. In all cases, the indicated acid concentrations were maintained reasonably constant throughout the test period, being controlled by titration at 3-hr. intervals with appropriate changes in the rate of solution feed. All meters were kept in calibration by frequent comparison with standards, and, in most cases, indicating, curve-drawing, and integrating ammeters were all used as checks upon one another.

The following units are employed throughout this paper: current density, amperes per square foot of cathode surface immersed; acid concentration, grams per liter; temperature, degrees Centigrade; weight of deposit, pounds per square foot of cathode immersed.

TABLE 2.—*Current Efficiencies in Zinc Electrolysis*

A.—Bully Hill Ores. May, 1915 (Hansen). (1000-amp. cells.) Av. temp., 28°. Current density, 10. Plotted in Fig. 2.

Acid 8.8		Acid 16.7		Acid 24.6		Acid 39.9	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
1.83	99.8	1.80	98.9	1.78	98.6	1.78	98.3
3.11	99.3	3.11	98.2	3.08	97.4	3.06	96.0
4.45	98.8	4.39	97.3	4.33	96.2	4.22	94.3
5.50	97.3	5.45	95.8	5.38	93.7	5.27	91.2

B.—Bully Hill Ores. May, 1915 (Hansen). (1000-amp. cells.) Av. temp., 30°. Current density, 10. Plotted in Fig. 2.

Acid 23.4		Acid 44.9		Acid 65.5		Acid 83.7		Acid 99.3	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
1.89	97.3	1.89	97.3	1.83	96.4	1.78	95.7	1.67	92.9
3.67	97.6	3.66	95.5	3.59	95.0	3.39	89.7	3.22	86.7
4.95	97.8	4.83	95.3	4.72	93.4	4.39	87.2	4.00	80.0
6.17	96.7	5.83	94.0	5.72	90.6	5.00	79.2	4.55	73.5
7.44	94.2	6.72	90.0	6.27	84.2	5.22	75.5	4.95	71.2

C.—Bully Hill Ores. June, 1915 (Hansen). (1000-amp. cells.) Av. temp., 30°. Current density, 15. Plotted in Fig. 3.

Acid 17.6		Acid 34.6		Acid 50.5		Acid 68.0		Acid 84.0	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
2.85	99.5	2.83	98.5	2.78	98.1	2.72	96.2	2.64	94.5
4.89	99.0	4.62	96.3	4.60	95.7	4.45	93.0		
6.32	97.0	6.22	93.2	6.00	91.0	5.72	86.5		

D.—Bully Hill Ores. October, 1915 (H. R. Hanley). (1000-amp. cells.) Temp. 28°. Current density, 25. Plotted in Fig. 4.

Acid 35.0		Acid 75.0	
Wt.	C. Eff.	Wt.	C. Eff.
0.41	99.6		
1.52	98.4	1.67	96.0
2.86	96.9	4.45	92.6
4.61	96.6	6.77	90.5
5.00	96.3	7.27	88.9

TABLE 2.—*Continued.*

*E.*—California Smelter Flue-dust. November, 1915 (H. R. Hanley). (1000-amp. cells.) Temp., 30–40°. Current density, 25. Plotted in Fig. 4.

Acid 35		Acid 71		Acid 102		Acid 126 (Zn = 12)	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
2.37	99.1	2.22	97.6	1.75	86.8	2.25	86.3
4.51	96.4	4.40	97.7	3.52	87.8	2.93	76.6
6.70	97.0	5.70	94.2	4.87	84.0	1.67	82.6
1.53	97.3	1.95	96.4	2.46	90.4	3.36	75.9
3.55	97.5	4.22	96.6	4.32	86.9	1.69	78.1
5.82	96.5	6.20	93.4	5.42	79.0	3.38	75.8

*F.*—Utah Concentrates. May, 1917 (Hansen). (200-amp. cells.) Temp., 30–40°. Current density, 25. Plotted in Fig. 5.

Acid 42		Acid 74		Acid 104	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
4.07	97.8	3.86	92.6	3.62	87.7

*G.*—Tasmanian Concentrates. August, 1917 (Hansen). (20-amp. cells.) Temp. 25–30°. Current density, 25. Plotted in Fig. 5.

Acid Concentration	Wt.	C. Eff.	Acid Concentration	Wt.	C. Eff.	Acid Concentration	Wt.	C. Eff.
19.3	2.25	94.6	89.0	3.19	93.5	98.9	2.73	93.2
36.1	2.30	96.6	91.8	3.15	92.4	104.0	2.73	93.5
54.2	2.31	97.1	91.9	3.13	91.8	105.5	2.73	93.3
72.9	2.30	96.6	91.7	3.12	91.3	120.3	2.61	88.9
93.8	2.25	94.6	105.0	3.02	88.9	136.8*	2.01	68.7

\*Zn = 8.

*H.*—Tasmanian Ore "A." September, 1917 (Hansen). (20-amp. cells.) Temp., 25–30°. Current density, 25. Plotted in Fig. 5.

Acid 89.0		Acid 94.5		Acid 96.0		Acid 120.8		Acid 138.6 (Zn = 12)	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
3.40	94.3	3.27	90.5	3.28	91.0	3.03	83.9	2.02	55.7



TABLE 2.—*Continued*

*I.*—Tasmanian Ore "B." September, 1917 (Hansen). (20-amp. cells.) Temp., 30°. Current density, 25. Plotted in Fig. 4.

Acid 93.6		Acid 90.8		Acid 91.6		Acid 94.7	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
1.59	90.0	3.19	89.4	4.86	88.5	6.43	86.9
1.59	91.0*	3.19	89.6*	4.86	89.0*	6.43	88.1*

\* Same data computed to 90 acidity (previous curves).

*J.*—California Flue-Dust. November, 1915 (H. R. Hanley). (1000-amp. cells.) Temp., 37°. Current density, 37.5. Plotted in Fig. 6.

Acid 62.8 (Nov. '15)		Acid 63.7 (Mar. '16)	
Wt.	C. Eff.	Wt.	C. Eff.
1.9	95.0	4.95	93.5
3.3	95.5	5.05	92.0
4.8	96.0		
4.9	95.8		
5.0	97.5		
5.0	98.0		
5.4	96.5		

*K.*—Utah Concentrates. November, 1917 (C. H. Ricker). (25-amp. cells.) Temp., 25–30°. Current density, 35. Acidity, 90. Wt., 3.1 in all cases. C. Eff., 93.2; 93.0; 94.3; 95.0; 90.3.

*L.*—California Flue-dust. November, 1915, to March, 1916 (H. R. Hanley). (1000-amp. cells.) Temp., 40–45°. Current density, 50. Plotted in Fig. 6.

Acid 96		Acid 94.4		Acid 123		Acid 123	
Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.	Wt.	C. Eff.
3.90	94.8	4.1	94.0	1.7	85.5	2.2	85.0
4.50	94.5	4.2	95.0	3.8	83.0	2.2	86.0
4.60	93.8	4.3	94.5	3.9	94.5	2.4	84.8
5.05	91.5	4.3	95.5	4.0	86.7	2.4	83.2
....	....	4.4	96.0	...	....	2.8	82.0
....	....	4.5	94.8	...	....	3.2	86.5

The data in Table 2 are plotted in Figs. 2 to 6 inclusive, the points in a given series being linked together for identification.

Figs. 7 and 8 represent the transposition to "Acid concentration" as the variable, the final curves representing current efficiencies at definite unit weights of deposit. In these plots, the points indicated are

obtained from the smoothed curves preceding. The particular unit weights used—3 to 5 lb. deposit per square foot of immersed cathode surface—fairly cover the range of present commercial practice.

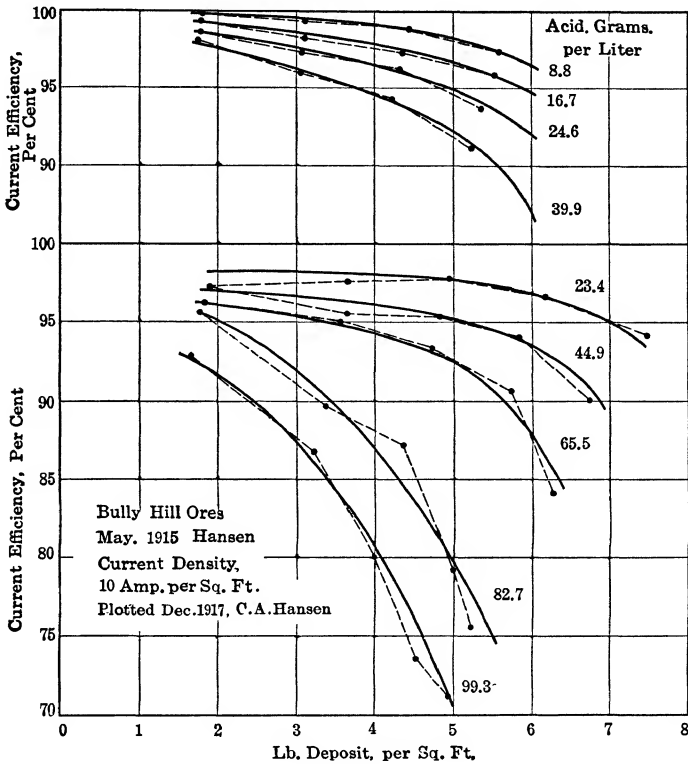


FIG 2.

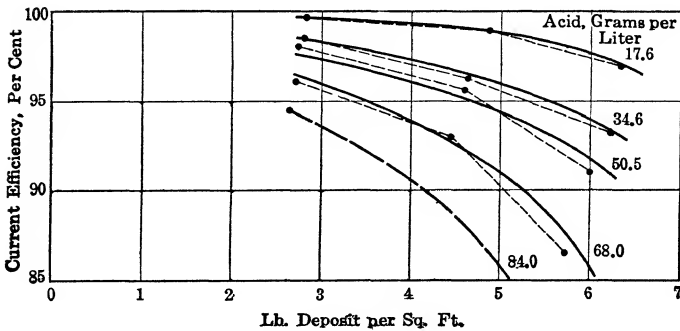


FIG. 3.—BULLY HILL ORES, JUNE, 1915. (Hansen.) CURRENT DENSITY, 15 AMP PER SQ. FT.

Figs. 9 and 10, in turn, serve to collect all of the preceding data on single curve sheets.

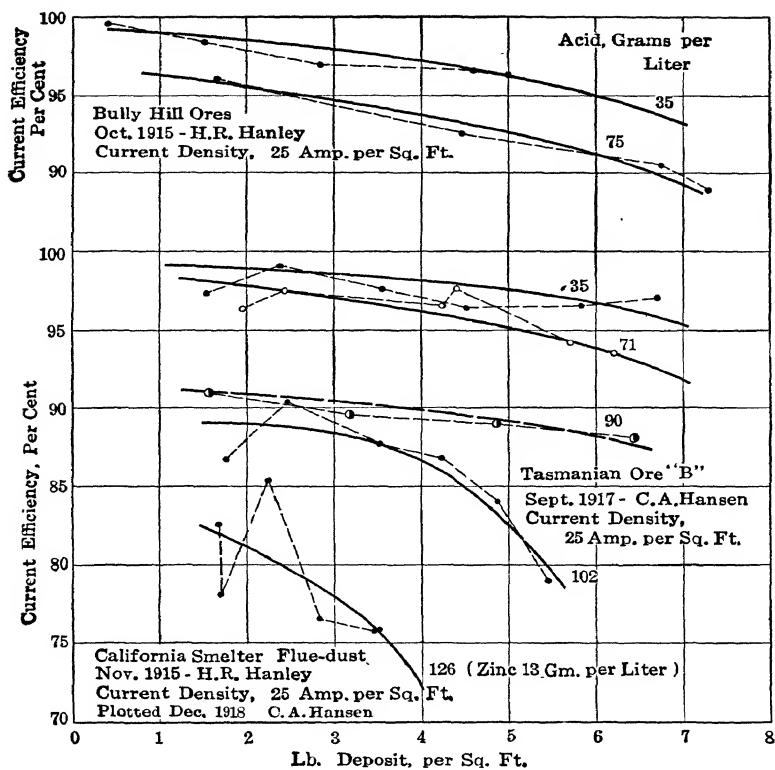


FIG. 4.

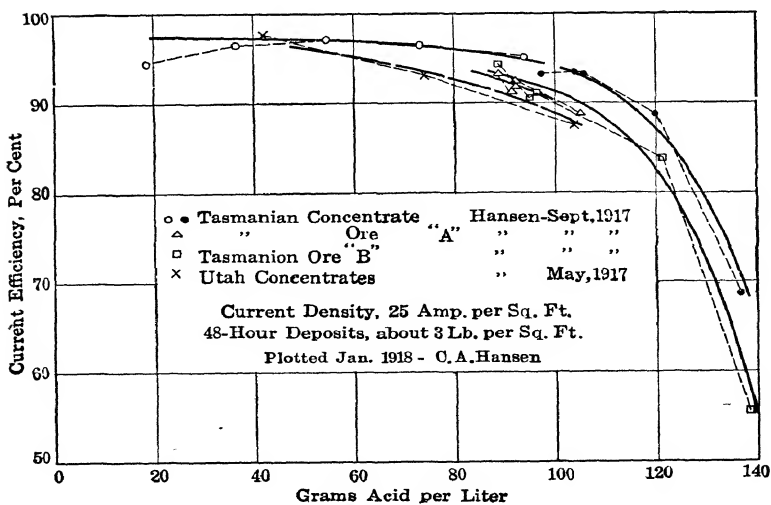


FIG. 5.

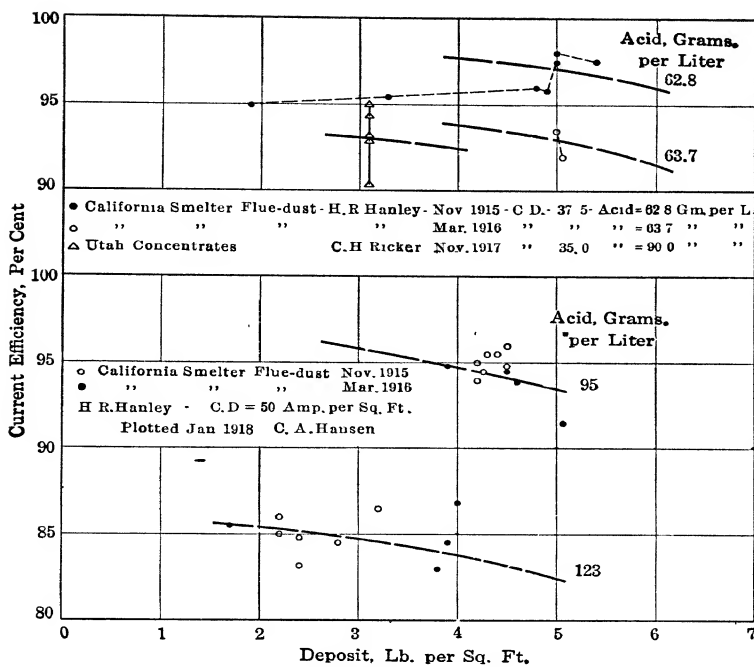


FIG. 6.

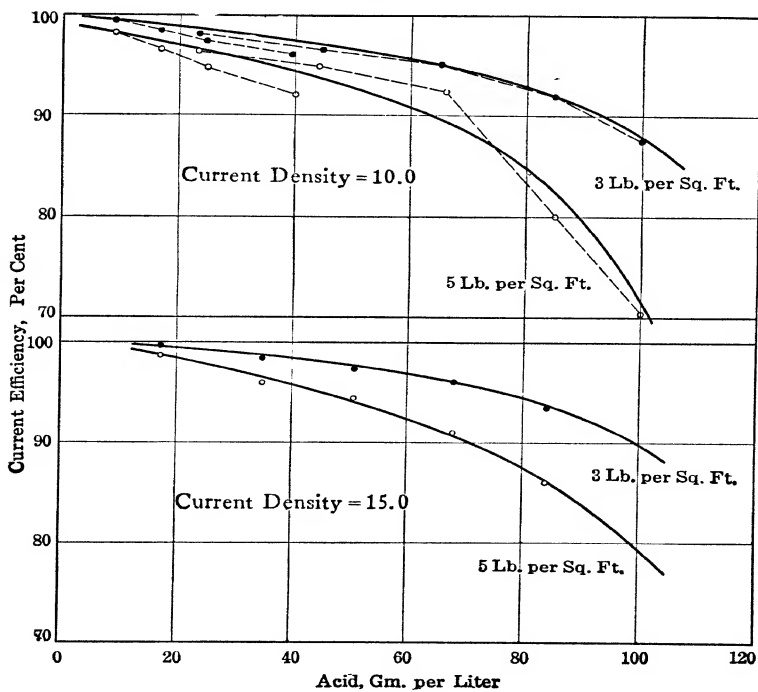


FIG. 7.

*Corrosion Rates*

According to the initial assumptions, the difference between the 100 per cent. current efficiency of deposition and the determined current efficiency is a measure of the corrosion rate. Corrosion rates, calculated from the data plotted in Figs. 9 and 10, are listed in Table 3. These are expressed in equivalent amperes per square foot for the sake of convenience.

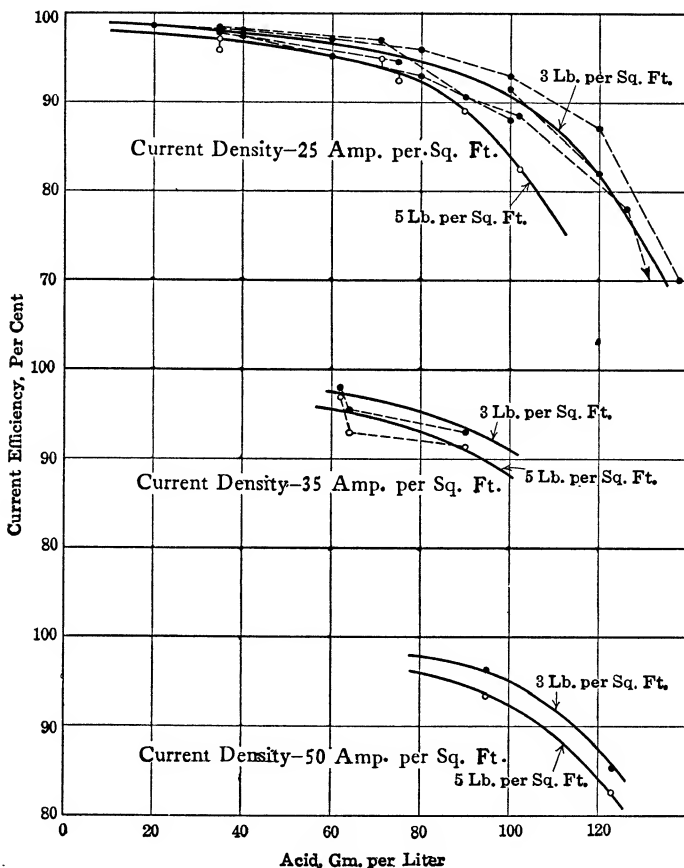


FIG. 8.

So far as these corrosion rates increase with the current density, they are in agreement with the observed facts that higher current densities do yield rougher deposits, particularly in the lower acid ranges. There does not, however, appear to be so marked a difference in the roughness of deposits made in high-acid electrolytes; in fact, all deposits made at above 100 gm. per liter acidity are reasonably smooth at all current densities (up to 100 amp. per sq. ft.) within the writer's experience.

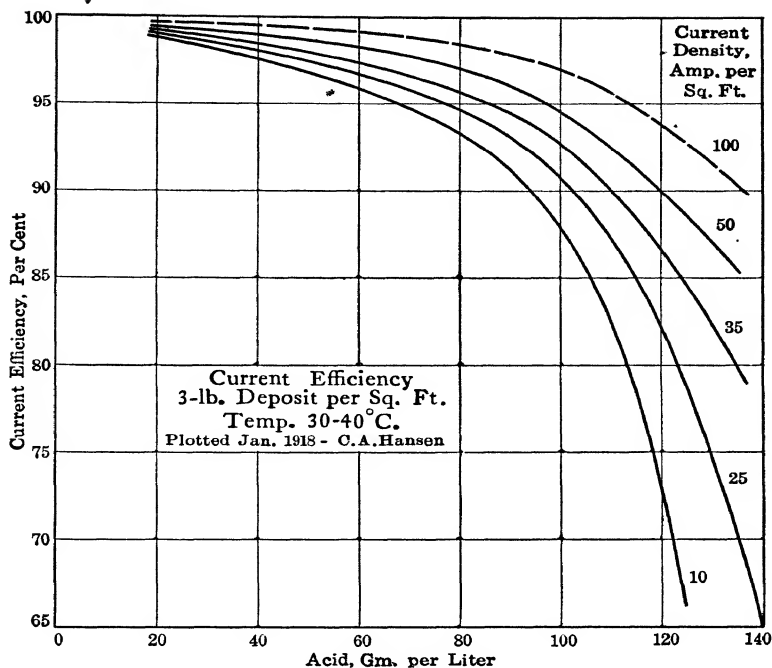


FIG. 9.

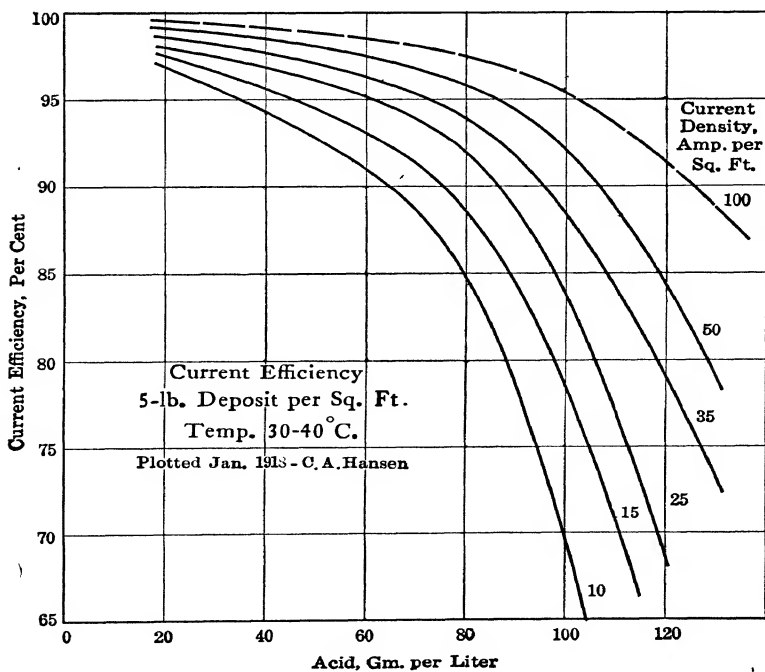


FIG. 10.

TABLE 3.—*Corrosion Rates in Equivalent Amperes per Square Foot*

Wt of Dep.	Current Density	Acid Concentration, Grams per Liter						
		20	40	60	80	100	120	140
3	10	0.12	0.25	0.43	0.67	1.22	2.70	
3	15							
3	25	0.17	0.50	0.82	1.34	2.35	4.50	8.75
3	35	0.28	0.56	0.81	1.89	2.69	4.72	8.05
3	50	0.30	0.55	0.90	1.50	2.75	5.10	8.15
5	10	0.30	0.57	0.90	1.52	3.00		
5	15	0.36	0.66	1.04	1.69	3.25		
5	25	0.50	0.80	1.20	2.05	4.05	7.90	
5	35	0.46	0.77	1.30	2.14	4.10	7.50	
5	50	0.45	1.00	1.25	2.10	4.00	7.90	
*	0	0.50	0.70	1.00	1.45	2.30	3.60	

\* Smooth deposits ( $\frac{1}{32}$ -in. thick) in cell solutions at zero current—from Fig. 1.

To make the observed corrosion rates agree with the initial assumptions would require that the 5-lb. deposit (at 120 gm. per liter acid) shall expose a surface equal to some two to three times its superficial area. It is highly improbable that it does so.

Zinc-sulfate electrolyte, however, gains in acid in proportion as it loses in zinc. Obviously, then, when the zinc becomes sufficiently low, the zinc concentration will have a greater effect upon the current efficiency than the acid concentration. It is further probable that the lack of zinc will be felt in proportion to the current density employed.

In what has preceded, the writer attempted to avoid zinc concentration as a factor in current efficiency by keeping to solutions containing a reasonable amount of zinc—20 gm. per liter or more—with the exception of two lower values specifically noted. Data regarding work with very low zinc concentrations were reserved for Table 4.

The comparison figures, extrapolated from Fig. 9, scarcely develop the desired or expected differences. It now appears more likely, considering that all of the higher-acid solutions had about the same initial zinc content (100 to 110 gm. per liter), that the data in Table 4 are only a natural extrapolation of the previously developed curve, and that the lack of zinc has at least a fairly important effect upon the current efficiency—an effect which becomes increasingly important towards the right (high-acid) side of the current efficiency diagrams. The major variable (note the corrosion rates at zero current density, and their agreement with the corrosion rates calculated from the current efficiency data, Table 3) is certainly acid concentration, and the final results are in very fair agreement with those that we should expect from the initial assumptions made.

*Voltage Required by the Zinc Cell*

The voltage data, presented in Table 5 and plotted in Fig. 11, were obtained in a specially constructed cell in which:

1. The lead electrodes were well peroxidized.
2. The electrodes were definitely parallel, and definitely spaced 2 in. apart ( $\frac{3}{16}$ -in. lead anodes,  $\frac{1}{8}$ -in. cathodes. Immersed cathode surface = 16 sq. ft.).
3. The electrolyte cross-section was the same in area as the immersed faces of the electrodes.

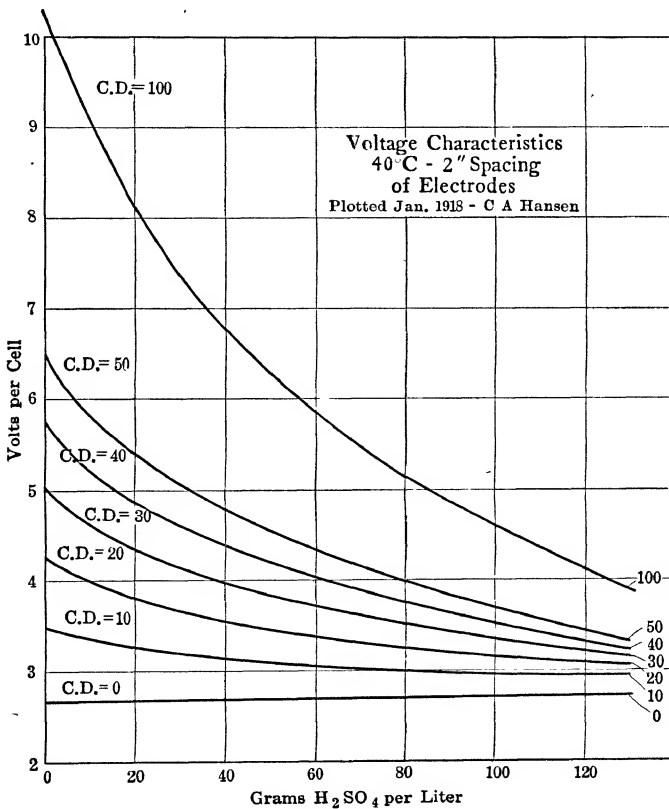


FIG. 11.

4. A lead steam coil served for temperature control, and an air lift for the circulation of solution to insure uniform temperature.

These precautions were taken in order to avoid the errors to which small cells are liable, most of which lead to underestimation of the required voltage. The figures given include contact and other losses.



TABLE 4.—*Electrolysis of Low-zinc Solutions*

Bully Hill Ores. November, 1915 (H. R. Hanley). (500-amp. cells.) Five successive strippings averaged.

Acid, grams per liter.....	144.00	148.00	157.00	166.00
Zinc, grams per liter.....	12.00	10.00	7 50	4.80
Temperature, °C.....	29.00	28.00	28.00	28.00
Current density.....	25.00	25.00	25.00	25.00
Pounds deposit per square foot..	1.49	1.21	1.02	0.54
Current efficiency, per cent.....	72.30	68.80	64 50	34.70
Comparative current efficiency extrapolated from Fig. 9.....	61.00	57.00	47.00	36.00

TABLE 5.—*Voltage at Different Temperatures*

Bully Hill Ores. June, 1915 (Hansen). Peroxidized lead anodes. Electrode spacing, 2 in.

Temp. °C.	Current Density							
	Acid, Grams per Liter	0	10	20	30	40	50	100
40	0	2.68	3.48	4 25	5.02	5 75	6.50	10 25
40	20	2.68	3 26	3.80	4.33	4.87	5.40	8.07
40	40	2.69	3.16	3.58	3.98	4 38	4.78	6.78
40	60	2.69	3.08	3.42	3.72	4.02	4.33	5 86
40	80	2.70	3.02	3.28	3.51	3.74	3.97	5.12
40	100	2.70	2.96	3.15	3.33	3.51	3.72	4.69
50	0	2.64	3.34	4.00	4.67	5.32	6.00	9.38
50	20	2.65	3.13	3.67	4.15	4.61	5.07	7.37
50	40	2.65	3.07	3.48	3.82	4.20	4.58	6.48
50	60	2.65	2 97	3.32	3.62	3.91	4.20	5.90
50	80	2.66	2.92	3 17	3.43	3.65	3.87	4.97
50	100	2.66	2.89	3.08	3.27	3.50	3.62	4.50
60	0	2.56	3.24	3.80	4.40	4.97	5.55	8.42
60	20	2.58	3.08	3.55	3.95	4.40	4.83	7.00
60	40	2.58	2.98	3.38	3.72	4.06	4.40	6.10
60	60	2.60	2 92	3.25	3.53	3.79	4.05	5.22
60	80	2.62	2.89	3.12	3.37	3.57	3.77	4.77
60	100	2.62	2.85	3.03	3.22	3.39	3.55	4.37
70	0	2.52	3.10	3.65	4.17	4.64	5.20	7.77
70	20	2.54	3.00	3.44	3.83	4.09	4.55	6.62
70	40	2.56	2.95	3.30	3.62	3.88	4.26	5.85
70	60	2.57	2.91	2.19	3.45	3.71	3.97	5.27
70	80	2.59	2.87	3.07	3.31	3.50	3.70	4.77
70	100	2.60	2.81	3.00	3.17	3.32	3.49	4.32

### Power Requirements of the Zinc Cell

Fig. 12 represents a summary of products of voltage and current efficiencies. Power is stated in terms of kilowatt-hours (direct current) required to deposit 2000 lb. of cathode zinc.

On melting the cathodes, the yield of ingot zinc averages 94 to 95 per cent. of the cathode weights charged.

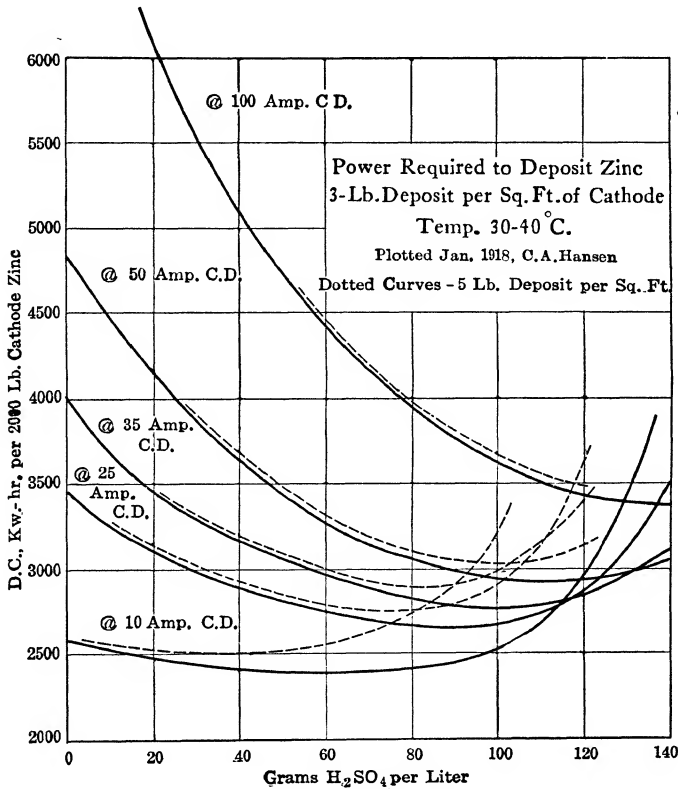


FIG. 12.

For converting alternating current (high-voltage) to direct current, the efficiencies of modern converting equipment may be taken at 92 per cent. for transformers and rotary converters, and 87 per cent. for transformers and motor-generators.

The direct-current data should, therefore, be multiplied by 1.145, or by 1.210, depending upon the choice of conversion apparatus, to compute the alternating-current requirements of the cell-room, per ton of ingot spelter, for the electrolysis of commercially pure zinc-sulfate solutions.

For the present, these data are offered without comment as to their

economic bearing. It may, however, be stated here that the only heat used in the leaching plant is that due to the solution of zinc oxide in the regenerated acid. High-acid concentrations bring about high leaching-tank temperatures; high leaching-tank temperatures conduce to excellent coagulation of the colloidal solids, and in that way make filtration possible. The amount of solution to be handled through the leach plant is also inversely proportional to the acid concentration of the leaching solution, and the zinc losses in the leach plant are practically proportional to the amount of solution handled.

It may also be stated that the cost of the cell-room is almost inversely proportional to the current density used, and that under any practicable conditions the cost of the cell-room and current-conversion equipment is a very reasonable fraction of the cost of the whole zinc plant.

Later, after publication of papers dealing with roasting, leaching, etc., when the various factors which enter into the economics of the art as a whole have been discussed, the bearing of the power data can be considered to better advantage than at present.

#### IMPURITIES IN ZINC ELECTROLYTE

The impurities that have been encountered in the course of our zinc investigation, not pretending that the list is complete, may be grouped into two fairly logical divisions:

1. Non-cumulative impurities; *i.e.*, such as are removed by the commoner purification methods in ordinary use, but likely to be present in the purified solution in proportion to the lack of thoroughness in purification.

2. Cumulative impurities; *i.e.*, such as pass clear through the entire plant in more or less undiminished concentration, and definitely accumulate in the closed solution circuit.

A great number of impurities known to be present in the raw materials, ores, concentrates, and reagents, have failed to materialize in the leach solution. These are dropped out of consideration entirely in this discussion, and we shall confine ourselves to the impurities which have been isolated from the solutions after they have left the leaching tanks.

The first group includes the following: copper, cadmium, arsenic, antimony, iron, manganese, silica, alumina.

The second group may be subdivided into strictly cumulative impurities which have no important bearing upon the problems of the zinc plant as a whole; and impurities which are injurious but are removable by specially devised purification processes.

The first sub-group includes the alkali metals and magnesium.

The second sub-group includes: nickel, cobalt, vanadium, possibly uranium.

*Non-cumulative Impurities*

All of the impurities listed in this group are widely distributed. Bully Hill ores were apparently entirely free from both arsenic and antimony, and it is probable that the Missouri zinc ores are likewise free of these impurities. With the exception of Bully Hill ores, arsenic and antimony have been present in all of the materials with which we have worked.

Iron, silica, and alumina are present in solution leaving the leaching tanks only so far as the solutions have not been properly oxidized and the colloids coagulated.

Manganese passes through the ordinary purification processes used in the leaching plant, but is thrown out in the cells as the insoluble dioxide.

Copper and cadmium are quantitatively precipitated upon metallic zinc. Arsenic and antimony are also precipitated on metallic zinc, but not so completely as to render the solutions practicable for use in the cell-room.

Chemical methods for the detection of these impurities are rather more sensitive than is the zinc cell to their presence in solution; hence it is not essential to rely upon the cell behavior to indicate their presence.

*Copper and cadmium* are precipitated electrolytically together with zinc, and when a cascade of cells is operated with solutions containing them, the current efficiencies obtained from the successive cells in the cascade are no longer consistent with the current-efficiency data ascertained with the use of pure solutions.

For example, the current efficiencies, based on 24-hr. deposits at 25-amp. current density, observed with a feed solution containing approximately 0.002 gm. Cu per liter, were as follows:

CELL	ACID, GRAMS PER LITER	C. EFF.
1	19.3	94.6
2	36.1	96.6
3	54.2	97.1
4	72.9	96.6
5	93.8	94.6

Copper was present in cells 1 and 2, but absent in cells 3, 4, and 5, having been deposited in the first two cells. Whether the precipitated copper alters the corrosion rate of the deposited zinc, or whether the copper in solution alters the activity of the solution, is still not clear. The characteristic of the above series, however, is thoroughly typical of such solutions.

Cadmium is probably less prejudicial to the behavior of the zinc cell than copper. This would follow from the relative positions of the two impurities in the electrochemical potential series. It is also indicated by the fact that some excellent deposits have been made which carried

as much as 0.33 per cent. Cd, while no satisfactory deposit of zinc has exceeded 0.03 per cent. Cu. The quantitative effects of these two impurities upon current efficiency, etc., are, however, of the same general order.

Copper and cadmium additions to solutions known to be otherwise satisfactory show the following current efficiencies (24-hr. deposits; 25-amp. c. d.; temp. 22–26° C.):

	Cu OR Cd 0	MILLIGRAMS PER LITER 5                      10	
Acid, 30 gm. per liter.....	99	93	90
Acid, 60 gm. per liter.....	96	87	81
Acid, 90 gm. per liter.....	93	76	65

*Arsenic and antimony* are apparently not precipitated electrolytically with zinc; at any rate, if they are present in solution the current efficiencies of the whole series of cells are materially reduced, and the impurities are still found in the effluent electrolyte.

It has often appeared that both arsenic and antimony are less prejudicial when manganese is absent than when it is present. In fact, in solutions containing manganese, it has consistently appeared that a variation between 0.001 gm. per liter and 0.0003 gm. per liter of Sb made the difference between impossible and excellent cell-room operation. It is obvious that the possible inter-reactions, when two or more impurities are considered, are almost infinite. Certainly, the data now at hand have scarcely opened this field for investigation. If it is really true that the manganese-antimony combination is particularly bad, it may very well be due to some cyclic reduction of antimony at the cathode and its oxidation by means of permanganic acid at the anode.

The highest current efficiencies obtained from solutions containing arsenic or antimony were observed in manganese-free solutions; the following table of current efficiencies indicates the effects of these impurities in manganese-free solutions (synthetic solutions; 24-hr. deposits; 25-amp. c. d.; temp. 22–26° C.):

Acid, Grams per Liter	Arsenic, Milligrams per Liter			Antimony, Milligrams per Liter		
	0	5	10	0	5	10
30	99	94	90	99	91	84
60	96	83	77	96	80	65
90	93	66	50	93	63	35

Some years ago, we attempted to purchase pure zinc sulfate for comparison standards. The best of the "cp" salts, when made up to 100 gm. zinc per liter with pure water, contained 40 to 50 mg. per liter of arsenic, about the same of antimony, and several times this amount of cadmium. Experiments with similar solutions, the chemical purity of

the salts being assumed from the label, may perhaps explain the obvious discrepancies in the literature regarding the electrolysis of zinc sulfate solutions. The purchased salts do give satisfactory results when they are purified by current methods of the zinc plant.

*Iron.*—The behavior of iron in solution is less consistent, as shown by the following table of current efficiencies observed with a solution containing: Mn, 0.50; Sb, 0.0016; Zn, 72.0 gm. per liter (current density, 25 amp.; 24-hr. deposits):

FE, GRAMS PER LITER	ACIDITY	C. EFF.	THEORETICAL C. EFF.
0.10	65	69	96
0.55	76	68	95
1.00	76	40	95

It is evident that iron in the electrolyte serves no good purpose. It was thought that ferric sulfate might conceivably act as depolarizing agent, absorbing the hydrogen which adheres to the cathode, and that it would be re-oxidized at the anode. This beneficent reaction apparently takes place, but zinc is dissolved as well as hydrogen. Cathodes made in solutions containing 1.0 gm. per liter Fe did not contain appreciably more iron than cathodes made in solutions carrying the normal 0.02 gm. per liter Fe.

*Manganese*, apparently, is the only element in the first group of impurities which, by itself at least, exerts no harmful influence. When manganese-bearing solutions are electrolyzed, the manganese sulfate is in part oxidized to permanganic acid at the anode. This permanganic acid probably reacts with manganese sulfate to throw out manganese dioxide. The reaction between permanganates and manganese sulfate is quantitative, and instantaneous, in hot, neutral (or basic), solutions. It is a comparatively slow reaction in cold, acid, solutions. We should therefore expect higher concentrations of permanganic acid when strongly acid electrolytes are in equilibrium with active cells.

On the other hand, this presumes that manganese dioxide is actually formed at some appreciable distance from the anodes, *i.e.*, by interaction of two reagents dissolved in the solution. If this is the case, there is no *a priori* reason why the manganese dioxide slime should always contain about the same percentage of lead peroxide. It appears, however, that the cell slime always does carry about the same ratio of manganese to lead, and that the loss of lead at the anode is more nearly proportional to the amount of manganese dioxide precipitated than it is to the amount of zinc deposited, or to the acidity of the solution.

The behavior of manganese dioxide at the anode in low-acid electrolytes also indicates that the dioxide is formed at the anode surface, since, when the acid is kept below 60 to 70 gm. per liter, the dioxide adheres to the anode and forms dense scales which appreciably add to the

electrical resistance of the cell. (It may raise the required voltage by 0.5 per cell.) With higher acidity, manganese dioxide does not adhere to the anodes at all, but falls to the bottom of the cell as a very finely divided slime.

The following results are typical of the behavior of manganese (25-amp. current density; temp. 30–35° C.). (Hansen.)

A.—Five cells in cascade; solution fed to cell No. 1 only. Analyses represent the condition of the solution in equilibrium with its active cell. All assays in gm. per liter.

	TOTAL MN	HMNO <sub>4</sub>	ACID
Feed solution.....	0.475	0.0000	0.0
Cell No. 1.....	0.332	0.000	18.7
Cell No. 2.....	0.243	0.0113	35.8
Cell No. 3.....	0.147	0.0280	55.3
Cell No. 4.....	0.090	0.0302	76.2
Cell No. 5.....	0.075	0.0310	95.2
Cell sump.....	0.075	0.0310	95.2

B.—Same cell arrangement; solution fed in equal amounts to cells 1, 2, and 3, and no direct feed of neutral solution to cells 4 and 5.

	TOTAL MN	HMNO <sub>4</sub>	Acid
Feed solution.....	0.737	0.000	0.00
Cell No. 1.....	0.197	0.027	88.9
Cell No. 2.....	.....	.....	91.7
Cell No. 3.....	0.170	0.034	93.7
Cell No. 4.....	.....	.....	120.8
Cell No. 5.....	0.080	0.030	139.3
Cell sump.....	0.080	0.030	139.3

C.—Same cell arrangement; solution fed in equal amounts to each of the first four cells.

	TOTAL MN	HMNO <sub>2</sub>	Acid
Feed solution.....	1.250	0.000	0.0
Cell No. 1.....	0.265	0.054	93.4
Cell No. 2.....	0.162	0.0544	92.8
Cell No. 3.....	0.147	0.0544	93.6
Cell No. 4.....	0.133	0.0544	92.6
Cell No. 5.....	0.063	0.0544	104.0
Cell sump.....	0.063	0.0544	104.0

The manganese dioxide slime combined from the above tests assayed: MnO<sub>2</sub>, 68.0; Pb, 13.7; Zn, 1.2 per cent. The anode loss amounted to about 6.9 lb. lead per ton of cathodes made.

The slime assay is fairly typical. The lowest lead content found in the dioxide slime was about 8 per cent.; this was derived from solutions containing 0.04 gm. per liter Mn, and the average lead loss at the anodes approximated 1 lb. per ton of cathodes made.

#### *Cumulative Impurities*

The impurities listed in the first sub-group are not at all important ordinarily. Magnesium is more likely to come from the lime used as

coagulent for the leach solution, than from the zinc ores. At Bully Hill, magnesium accumulated until it reached about 12 gm. per liter without having any appreciable effect upon the cell behavior. Soda and potash have been found to the extent of 4 gm. per liter in solutions that had been circulated rigidly for long periods.

In the second sub-group are found the insidious impurities, the only ones of which we know at present. In general, the cells are more sensitive to them than the chemical methods as yet evolved for their estimation or detection.

As the solutions are circulated rigidly through leach plant, cell-room, and leach plant again, these impurities definitely accumulate, and their ill effects mount up as indicated in the following table of current efficiencies. The five cells were cascaded, and only No. 1 cell was fed with neutral solution. Successive strippings are recorded.

July–August, 1916. (Hansen). 24-hr. deposits; 25-amp. per square foot; temp. 30–40° C.

Acidity, Grams per Liter	14	26	37	48	58
C. eff. No. 1, Stripping ...	93.8	89.2	90.2	89.7	69.4
C. eff. No. 2, Stripping ...	90.5	81.6	77.8	56.0	35.9
C. eff. No. 3, Stripping ...	85.8	69.4	76.8	....	42.7
C. eff. No. 4, Stripping....	83.5	..	39.3	27.9	29.3
Pure Sols. c. eff. (Fig. 9).	99.2	98.8	98.2	97.5	96.7

*Cobalt*.—It developed that in the above work about 4 mg. per liter of cobalt was added at each leaching cycle (0.002 per cent. Co based on the calcine leached). The behavior of cobalt is characteristic of the behavior of all the members of the group.

Synthetic manganese-free solutions electrolyzed at 25 amp. c.d. to yield 24-hr. deposits at 25° C., indicate the following quantitative effects of cobalt on current efficiency:

Co, Milligrams per Liter	0	5	10	15	20
Acid 30 gm. per liter.....	99	96	93	91	87
Acid 60 gm. per liter.....	96	90	85	77	64
Acid 90 gm. per liter.....	93	80	70	55	30

Cobalt has been found in Australian concentrates, and in the Coeur d'Alene zinc ores. It is reported in the ground waters of the Missouri zinc field, and in the ground waters of the Bawdwin (Burmah) mines. It is possible that its distribution is rather widespread.

*Vanadium* has been found to give similar troubles, having been encountered in Leadville zinc ores. It has been detected spectroscopically



in Utah ores, but for this work sufficiently accurate methods have not been developed to detect vanadium by chemical methods, although it is probably often present to an extent sufficient to make very serious trouble.

### *Impurities in General*

Pure solutions invariably yield excellent deposits, which are wholly without blemish on the reverse, or starting-sheet, side.

Impure solutions invariably yield imperfect cathodes. Pinholes, much more readily seen on the starting-sheet side of the cathode, are almost always found when the merest traces (tenths of milligrams per liter of electrolyte) of arsenic, antimony, nickel, cobalt, or vanadium are present. Curiously, if any considerable quantity of any of these impurities is present, the holes in the cathode are enlarged at the starting-sheet side of the deposit, giving the appearance of having started with local corrosion at the starting-sheet surface and then corrosion working its way toward the surface directly exposed to the action of the solution.

When serious quantities of impurity are present, deposition may proceed in excellent shape for several hours; the deposit may then corrode very rapidly until it entirely disappears in the course of a few hours; when it has entirely dissolved, perhaps exposing the clean starting sheet only in patches, deposition may resume in good shape. This cycle of alternate deposition and dissolution may be repeated more or less automatically as long as one chooses.

The nature of the reactions causing corrosion is still undetermined. Corrosion may result from deposition of an impure zinc from an impure solution, the impurities in the zinc itself altering the cathode's resistance to corrosion. That an impure zinc does corrode much more rapidly than a pure zinc is evidenced by the following observations on three samples of zinc which were suspended side by side in each of several cascaded active cells.

	[Loss, Grams Per Square Foot Per 24 Hr.		
	A	B	C
Acid, 108 gm. per liter.....	2.7	135.5	271.0
Acid, 84 gm. per liter.....	1.35	37.0	129.5
Acid, 63 gm. per liter.....	1.35	6.2	49.3
Acid, 41 gm. per liter.....	0.0	.....	44.3
Acid, 21 gm. per liter.....	.....	6.7	22.0
Assays, per cent.:			
Cu.....	0.005	0.002	0.005
Cd.....	0.030	0.080	0.020
Fe.....	0.005	0.0385	0.030
Pb.....	0.040	0.1367	0.060

Sample A, unmelted cathode zinc, good deposit.

Sample B, M. & H. rolled sheet zinc.

Sample C, good cathode deposits melted in iron pots, and sheets sawed from the resulting slabs.

The above data, at any rate, indicate that the purity of the zinc can make differences of 100 : 1 in corrosion rates.

On the other hand, since zinc is electropositive to all of the impurities with which we have been concerned, if one collected the last remains of a rapidly corroding deposit, one should expect to find the impurities concentrated in that fraction. Yet, even when only a black scummy residue is all that is left of the deposit, that scum is invariably lead and copper. The causative impurities are not detectable, or only barely detectable, in the residue.

#### TEMPERATURE AND STABILITY OF ZINC CELL

When dealing with the effects of temperature within the zinc cell, the matter necessarily is highly speculative since few data bearing specifically upon this point are as yet available. In the case of pure solutions, and the writer firmly believes that working with any other than pure solutions is out of question commercially, the effects of cell temperature are apparently not important.

The whole discussion rests on the observation that the zinc cell appears to be essentially stable with pure solutions, and unstable with impure solutions. It appears that low current efficiencies produce high cell temperatures; that the high cell temperatures induce increased corrosion (and still lower current efficiencies), and so on.

Since the quantitative relationship between corrosion and temperature appears not to have been studied, the major variable in this proposed discussion is an unknown quantity. Chemical reactions may, however, be grouped in fairly well defined families, the characteristics of which appear to vary in the same general order when one of the conditions is altered. For example, the student chemist is taught that reaction rates between dissolved substances usually double with each rise of 10° C. in temperature.

Addicks reports the behavior of copper in acid copper sulfate and in acid ferric sulfate-copper sulfate electrolyte. In this connection Addicks also assumes that current efficiency is determined by corrosion rates, and gives the following data on the corrosion of copper as a function of temperature.

#### *In Acid Copper Sulfate*

UNITS CORROSION	°F.
0.5	87
1.0	100
2.0	117
4.0	142
8.0	168

In acid copper sulfate, the reaction rate doubles (on the average) with each 20.5° F. rise in temperature (11.4° C.).

*In Acid Ferric Sulfate-Copper Sulfate*

1 Per Cent. Fe		2 Per Cent. Fe		3 Per Cent. Fe	
Cor.	Temp., °F.	Cor.	Temp., °F.	Cor.	Temp., °F.
0.0200	78	0.040	78	0.062	78
0.0370	123	0.070	123	0.110	123
0.0800	167	0.160	167	0.270	167

In acid ferric sulfate-copper sulfate, the reaction rate doubles (on the average) with each 40° F. rise in temperature (22° C.).

Unfortunately, these two sets of data (which were assumed to cover similar reactions having the same temperature coefficient) do not check very satisfactorily. If we take the greater temperature interval, however, and apply it to the corrosion of zinc, the choice works against the following argument in that its conclusions become less spectacular and, therefore, perhaps safer.

Accordingly, by assuming that the corrosion rate of cathode zinc doubles with each 22° C. rise in the temperature of the cell solution, and referring all corrosion rates to the corrosion rate at 40° C. as unity, the temperature-corrosion relationship becomes:

TEMP., °C.	CORROSION RATE	TEMP., °C.	CORROSION RATE
25	0.58	55	1.61
30	0.70	60	1.88
35	0.82	70	2.60
40	1.00 arbitrary unit	80	3.56
45	1.17	90	4.80
50	1.37		

Addicks<sup>1</sup> publishes a curve showing the relation between the energy dissipated as heat within the copper refining cell and the temperature maintained when the cell is in equilibrium with these losses. For practical purposes, the copper cell is identical with the zinc cell in its facilities for dissipating its heat energy. (The zinc cell will dissipate slightly more heat at a given temperature than the copper cell because the anode oxygen liberated in the zinc cell increases evaporation somewhat.)

<sup>1</sup> Lawrence Addicks: Current Density in Copper Refining. *Metallurgical and Chemical Engineering* (Mar. 15, 1917), 16, 311.

Addicks' cell offered 312 sq. ft. of active cathode surface, and would therefore take:

CURRENT DENSITY	AMPERES PER CELL
10	3,120
25	7,800
50	15,600
100	31,200

The energy losses bore the following relation to cell temperature:

KILOWATT LOSS PER CELL	CELL TEMP. (°C.)	KILOWATT LOSS PER CELL	CELL TEMP. (°C.)
1.70	30	8.00	55
2.55	35	10.00	60
3.60	40	15.00	70
4.85	45	22.00	80
6.30	50	30.00	90

{ Extra-  
polated

In the copper cell, no chemical changes occur which absorb energy, and all of the electrical energy supplied must be dissipated as heat.

In the zinc cell, the zero current potential is 2.68 volts per cell (Helmholtz equation gives the value 2.31 calculated from the heats of reaction involved) and this potential of 2.68 corresponds to the absorption of 2000 kw.-hr. per ton as necessary for the deposition of 2000 lb. of cathode zinc at 100 per cent. efficiency. The difference between the above 2000 kw.-hr. and such power as is actually consumed in the cell is then a measure of the energy which the cell must dissipate as heat, by evaporation (Antisell states that 90 per cent. of the cell energy is accounted for by evaporation of electrolyte in the copper refinery), by radiation, by convection, etc.<sup>2</sup>

It is obvious that equilibrium cell temperature corresponds to that temperature at which the energy losses within the cell exactly equal the capacity of the cell for dissipating that energy. If the energy losses within the cell increase, with rising temperature, more rapidly than the capacity of the cell for dissipating that energy, the cell is essentially unstable.

For the purpose in hand, we shall analyze only a very few cell conditions. We shall assume an acid concentration of 90 gm. per liter, pure solutions, and a few different current densities, for the purpose of indicating the effect of current density upon cell temperatures, adopting a cell of the size and capacity of Addicks' copper cell.

<sup>2</sup> With reference to the heating of solutions within the cell:—A 10,000-amp. cell ordinarily requires about 3.5 liters of solution per minute. Each degree (C.) rise in solution temperature accounts for 0.2441 av. Ordinarily the temperature rise is not sufficient to make it an important item in the cell heat balance sheet.

A.—C.d., 10 amp. per square foot; acid, 90 gm. per liter; pure solutions; deposits, 3 lb. per square foot; current per cell, 3120 amp. Neutral solution feed. Temp. solution feed, 30° C.

TEMP., °C.	30	40	50
Volts per cell.....	3.10	2.99	2.90
Kilowatts per cell.....	9.67	9.33	9.05
Corrosion rate.....	0.70	1.00	1.37
Current efficiency, per cent....	93.7	91.0	87.6
Zinc made, pounds per hour..	7.85	7.62	7.32
Solution required, l. per hr...	59.6	57.9	55.7
Kw. acct'd for by zinc .....	7.85	7.62	7.32
Kw. acct'd for heating solution	0.00	0.67	1.29
Kw. cell losses (difference)...	1.82	1.04	0.44
Cell can dissipate, kilowatts..	1.70	3.60	6.30

Equilibrium temperature = 31 0° C.

B.—C.d., 25 amp. per square foot; acid, 90 gm. per liter; pure solutions; deposits, 3 lb. per square foot; neutral solution feed at 30° C.; current per cell, 7800 amp.

TEMP., °C.	40	50	55	60
Volts per cell.....	3.32	3.23	3.20	3.17
Kilowatts per cell.....	25.9	25.2	24.9	24.7
Corrosion rate.....	1.00	1.37	1.61	1.88
Current efficiency, per cent.....	93.0	90.4	88.7	86.8
Zinc made, pounds per hour.....	18.7	18.2	17.8	17.4
Solution required, l. per hr.....	148.00	144.00	141.00	138.00
Kw. acct'd for by zinc.....	18.7	18.2	17.8	17.4
Kw. acct'd for heating solution...	1.7	3.3	4.1	4.8
Kw. cell losses (difference).....	5.5	3.7	3.0	2.5
Kw. cell can dissipate.....	3.6	6.3	8.0	10.0

Equilibrium temperature = 44.2° C.

C.—C.d., 50 amp. per square foot; acid, 90 gm. per liter; pure solutions; deposits, 3 lb. per square foot; current per cell, 15,600 amp. neutral solution feed at 30° C.

TEMP., °C.	40	60	70	80
Volts per cell.....	3.85	3.65	3.55	
Kilowatts per cell.....	60.0	57.0	55.4	(53.8)
Corrosion rate.....	1.00	1.88	2.60	3.56
Current efficiency per cent.....	96.0	92.5	89.6	85.8
Zinc made, pounds per hour.....	39.9	38.5	37.3	35.7
Solution required, l. per hr.,....	305.00	295.00	285.00	273.00
Kw. acct'd for by zinc.....	39.9	38.5	37.3	35.7
Kw. acct'd for heating solution...	3.5	10.3	13.2	15.9
Kw. cell losses (difference).....	16.6	8.3	4.9	2.3
Cell can dissipate, kilowatts.....	3.60	10.0	15.0	22.0

Equilibrium temperature = 58° C.

*D.*—C.d., 100 amp. per square foot; acid, 90 gm. per liter; pure solutions; neutral solution feed at 30°C.; deposits, 3 lb. per square foot; current per cell, 31,200 amp.

TEMP., °C	40	70	90
Volts per cell.....	4.90	4.55	
Kilowatts per cell.....	153.0	142.0	(135)
Corrosion rate.....	1.00	2.60	
Current efficiency per cent.	97.7	94.0	
Zinc made, pounds per hour	81.7	78.6	
Solution required, l. per hr.	605	582	
Kw. acct'd for by zinc.....	81 7	78.6	(75.0)
Kw. acct'd for heating solution.....	7 0	27 0	(38.6)
Kw. cell losses (difference)..	64 3	36 4	(21.4)
Cell can dissipate, kilowatts	3 6	15.0	(30.0)

Equilibrium temp. at approximately 85°C.

In any of the above cases it appears that the zinc cell is essentially stable, that is, the capacity of the cell for dissipating heat rises very rapidly with increasing temperature while the internal cell losses decrease with rise in temperature within the ranges involved:

Suppose, however, that the initial corrosion rate is already high. Is it not then possible that the internal energy losses of the cell mount more rapidly with temperature than the capacity of the cell for ridding itself of heat?

For the purpose of this argument, we can assume that we have a solution so impure that we can get a current efficiency of only 70 per cent., at 25-amp. c.d., with 90 gm. per liter acidity and at 30° C., instead of over 96 per cent., as we should from pure solutions. Such a solution is by no means an absurdity when the leach plant fails to operate properly, or an odd lot of purchased ore is thrown into the plant without investigation of its antecedents. It has been shown that a very few milligrams of any one of several impurities—arsenic, antimony, cobalt, vanadium, etc.—will cause greater reductions in current efficiency than that assumed.

Amperes per cell, 7800 (C.d. 25 amp per sq. ft.)			
TEMP., °C.	30	50	70
Volts per cell.....	(3.40)	3.25	3.13
Kilowatts per cell.....	26.5	25.2	24.4
Corrosion rate.....	0.70	1.37	2.60
Current efficiency, per cent.	70.0	41.3	(-10)
Zinc made, pounds per hour	14.6	8 8	0.0
Solution required, l. per hr.	115.00	69.00	0 00
Kw. acct'd for by zinc.....	14.6	8 8	0.0
Kw. acct'd for heating solution.....	0 0	1 6	0.0
Kw. cell losses (difference)..	11.9	14 8	24.4
Kw. cell can dissipate.....	1.7	6.3	15.0

Equilibrium temperature, 84° C. The equilibrium condition is also one at which no zinc is deposited at the cathode.

The conclusion drawn from this temperature argument is thoroughly in keeping with the observed behavior of zinc cells operating with impure electrolyte.

*Practical Expedients for Control of the Zinc Cell*

When a condition arises in the commercial cell-room which leads to the results above described, there are two ways of compromising with it, neither of which is very satisfactory. Perhaps the better one is to flush out the bad electrolyte as rapidly as possible. This requires very liberal design of the leaching plant in order that it shall not be flooded by the return of large volumes of solution which it must re-treat.

The second method is to cut down the cell current, while maintaining the feed. This serves two purposes, in that the acid concentration falls off and so reduces the corrosion rate; and, the total energy supplied to the cell being less, the heat which the cell must dissipate falls off correspondingly. This lowers the cell temperatures, and the corrosion rate is further decreased. Also, as the current is diminished—while it may readily result in an increased production of spelter for the given conditions—still the output of spelter for the plant becomes outrageously low.

There is a third method which can be used—and frequently is—to some advantage. Glue, lanolin, and perhaps a number of other organic reagents, when added under such conditions, do materially increase the current efficiency. The effect appears to be only temporary, however, even when the added reagent is fed to the electrolyte in each cell independently and constantly.

The effect of these addition agents has been studied to a considerable extent, and their use is unquestionably of some benefit. It is, however, so firmly the writer's opinion that the addition of such reagents is but a poor makeshift corrective for unpardonably lax leaching, that the discussion of their effects is deliberately avoided in order that the primary conclusions of this paper shall not be obscured.

The conclusions of this paper are based upon normal market conditions. At the present time, certain reagents, notably the permanganates, of which the zinc plant is desperately in need for its purification processes, are not commercially available. In these circumstances, probably every one connected with electrolytic zinc work is forced to compromise as best he can. In so far as he realizes that he is compromising with abnormal economic conditions, and that he is not merely making shift to cover up conditions that can be otherwise corrected, he has the writer's entire support and heartfelt sympathy.

ACKNOWLEDGMENTS

The data contained in the foregoing paper are based entirely upon the work of the writer and his associates at Bully Hill, and the work in

a small experimental plant which Mr. G. W. Lambourne (Manager of the Judge M. & S. Co.) permitted the writer to install, and experiment with, at the Judge zinc plant at Park City, Utah. The writer very gladly acknowledges the excellent and valuable work of J. B. Keating, H. R. Hanley, E. M. Baldwin, H. F. Bradley, K. P. Girard, and of the various others who have at one time or another been with us in this work during the past several years.

#### APPENDIX\*

Since the above paper was written, tests have been conducted with a view to securing a sounder basis for discussing the effects of temperature on the behavior of the zinc cell. The following data, while making no pretense to great accuracy, are still reasonably close.

Cells were operated with the same feed solutions, but at varying temperatures, the latter being controlled by means of immersed steam coils. Current efficiencies obtained indicate that the corrosion rate doubles with each rise of 21.7° C. in electrolyte temperature. No great error is therefore introduced in the original paper as a result of the temperature coefficient there assumed.

Strangely enough, parallel tests conducted by merely suspending cathode sheets in solutions maintained at different temperatures (the sheets not functioning as cathodes at all) indicated no definite temperature coefficient of corrosion. It appeared in these latter tests that adhering hydrogen interfered with the free access of the acid solutions to the zinc surfaces.

Tests were also conducted with an electrically heated evaporating pan (immersed heaters) in which some 2 sq. ft. of electrolyte surface was freely exposed to the cell-room atmosphere. The data obtained, corrected so as to cover merely losses of energy from the actual solution surface, indicate surface losses as follows (room temperature 20 to 25° C.):

SOLUTION TEMP., DEG. C.	WATTS LOSS PER SQ. FT.	SOLUTION TEMP., DEG. C.	WATTS LOSS PER SQ. FT.
30	25	50	155
35	45	60	270
40	70	70	400
45	115	80	540

These data are very closely checked by an independent set of tests conducted on commercial cells.

In the temperature range 40 to 45° C., evaporation of solution from electrolyte surface accounted for 87 per cent. of the energy dissipated at the surface, this figure being in excellent agreement with the 90 per cent. figure quoted from Mr. Antisell.

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\* Received Oct. 4, 1918.



I think it probable that the energy lost from the cell solution surfaces is mainly accounted for by evaporation at all temperatures, since the relation between vapor pressure of the electrolyte and the energy losses determined is too simple to be mere coincidence.

## DISCUSSION

J. L. MCK. YARDLEY,\* Pittsburgh, Pa. (written discussion†).—It is interesting to observe how closely Mr. Hansen agrees with other investigators to the effect that the art of electrolytic zinc has left the realm of mystery. In his introduction, he has said practically what Thomas French said in his paper "The Future of Electrolytic Zinc."<sup>1</sup>

The successful electrolysis of zinc from sulfate solutions has not been an easy matter, and it is only within recent years that the difficult problem of depositing high-grade zinc has been satisfactorily accomplished on a commercial basis. The principal requisite is that the electrolyte shall be quite free from certain impurities. The methods by which freedom from these impurities is assured are now very well understood, and with experienced superintendents there is little difficulty in obtaining a high efficiency in that part of the process.

When certain underlying principles are recognized, the difficulty encountered in dealing with the filtration of the acid and slimy solutions also largely disappears, although it has been a very serious one to the uninitiated. In the dissolving of the zinc from the roasted ore, there is nothing which any competent chemical engineer cannot undertake, and any other operations connected with the electrolytic process are either of little or no difficulty, or are common to the retort process.

Mr. Hansen also agrees with R. G. Hall's statement in "Some Economic Factors in the Production of Electrolytic Zinc:"<sup>2</sup>

The requirement, and I use it in the singular, for the electrolysis of zinc is zinc sulfate, and zinc sulfate only. Most other elements found in the solution are harmful to the electrolysis of zinc sulfate. I have never found any that I could confidently say were beneficial. This requirement seems relatively simple, but when it is understood that this process is to be applied to such a complex of ore minerals as is indicated above in the review of ore deposits, it will be understood that the production of zinc sulfate, pure, of the standard strength, is not the easiest problem in the world. And yet it is only this problem that has stood in the way, for all time past, of the manufacture of zinc electrolytically. All of the factors necessary to the production of a solid coherent plate of electrolytic zinc were known long ago, but it is only recently that we have been able to produce this pure zinc sulfate on a commercial basis and in large quantities, and have been able to obtain the electric current at such a cost as to make the production of electrolytic zinc a commercial possibility.

While it is doubtful that these gentlemen would agree concerning the most desirable methods for carrying out all the various parts of the process, it is noteworthy that they agree concerning the essentials in-

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† Received Aug. 10, 1918.

<sup>1</sup> *Transactions*, American Electrochemical Society (1917) **32**, 321.

<sup>2</sup> *Trans.* (1918) **57**, 709.

volved. Perhaps their differences regarding details would not be so much of fact as of experience. The important point is that there are at least three plants in the United States at which electrolytic zinc is being produced commercially from ores of widely different origin and composition, by processes the developments of which have been practically contemporaneous and independent.

### *Roasting Problem*

Both Mr. Hall and Mr. French have given us rather more than a hint as to what the problem in roasting has been. The particular factor that interfered with the highest recovery when the roasted ferruginous ore was leached with sulfuric acid was the formation, in the roaster, of a complex compound of zinc and iron which is insoluble. It developed that the production of this material was largely due to roasting at too high a temperature. Mr. Hall mentioned that, "In roasting Colorado ores for the production of spelter, the writer has found as much as one-third of the total zinc in such ore to be insoluble in a relatively strong solution of sulfuric or hydrochloric acid." Mr. French stated:<sup>3</sup>

With proper adjustment of the roasting furnace conditions, it is not difficult to obtain extractions of 90 per cent. of zinc from ores containing 12 to 25 per cent. zinc, and in the latter case with as much as 25 per cent. of iron. As far as the author's experience goes, furnaces of the Wedge and Herreshoff type are admirably adapted to the roasting of this class of ore. A large Wedge furnace, with seven hearths and a diameter of  $22\frac{1}{2}$  ft. (6.8 m.) is capable of roasting about  $1\frac{1}{4}$  tons of this ore per hour, with unskilled labor, and gives a more satisfactory product than the smaller hand-rabbed furnaces. After roasting zinc concentrates containing 45 per cent. of zinc, there is little difficulty in extracting as much as 95 to 97 per cent. of the zinc.

There is every indication, therefore, that exceedingly high recoveries should be obtained from Montana ores which contain around 22 per cent. of iron and are readily concentrated to exceed 50 per cent. of zinc. The general impression seems to be that the proper roasting temperature for subsequent leaching with sulfuric acid is around 600° C. I have understood for nearly a year now that this is the approximate temperature employed in the Wedge roaster at the Park City, Utah, smelter, working on oil flotation concentrates of around 40 per cent. zinc.

E. H. Hamilton, in a paper<sup>4</sup> on "Electrolytic Zinc Extraction at Trail, B. C.," stated the temperature of the Wedge roaster to be approximately 600° C. The ore had approximately the following composition:

	PER CENT.		PER CENT.
Lead.....	14	Magnesia.....	2
Iron.....	21 to 31	Sulfur.....	24 to 29
Insoluble.....	4	Zinc.....	19 to 24
Alumina.....	3	Cadmium.....	0.04
Lime.....	2		

<sup>3</sup> *Op. cit.*, 322.

<sup>4</sup> *Transactions*, American Electrochemical Society (1917) **82**, 217.

The ore is crushed in tube-mills to the following size:

MESH.	PER CENT.
On 48...	3
On 48-100...	10
On 100-150...	3
On 150-200...	12
Through 200...	72
	<hr/> 100

and is then roasted in Wedge roasters having seven floors and diameter of 26 ft. (8 m.) with two arms to the floor and revolving once in 4 min. Forty tons are roasted per day from 25 per cent. down to 5 per cent. sulfur. The temperature of the hearths is approximately:

1st floor..	.....
2d floor..	..... 950° F. (510° C.)
3d floor.....	..... 1000° F. (538° C.)
4th floor ..	..... 1100° F. (593° C.)
5th floor..	..... 1100° F. (593° C.)
6th floor.....	..... 1050° F. (566° C.)
7th floor.....	.....

Since the problem of roasting for subsequent leaching with sulfuric acid is the same everywhere, that is to eliminate sulfides and convert the zinc into an oxide or sulfate, it follows that the practice must be substantially the same everywhere, the time of the operation varying only with the fineness of the ore and the sulfur contents.

#### *Leaching and Purification Problem*

On the other hand, this problem differs with each locality. On visiting the successful electrolytic zinc plants of the country, one finds no lack of understanding of the impurities that occur, of the reagents which must be employed, or of the reactions that take place. While in some cases, past failures in leaching for electrolytic zinc were probably due to lack of consideration for things which were known, the difficulties encountered have been usually the more or less inevitable result of the large scale on which it has been attempted to operate. They have been attributable mainly to poor arrangement of the plant and to failure of some of the mechanical apparatus to operate continuously, particularly that connected with the filtration of the solution and the removal of the coagulated or precipitated impurities. It has been found absolutely essential to have a well designed plant, in which the apparatus is arranged in sequence according to the sequence of operations; in which lead pipe, and not iron or other substitute, is used where lead should be used; in which excessive pumping is avoided; in which reliable mechanical apparatus is employed and no one piece is so situated that it can become the solitary neck of the bottle, the temporary clogging of which might

cause the solution to foul and so disrupt the operation of the entire plant as to require days for its resumption.

### *Power Requirements of the Zinc Cell*

These requirements also are well understood, though they were not two or three years ago when most of the existing electrolytic zinc plants were planned. It was not appreciated that electrolytic zinc precipitation would become substantially a constant-voltage, constant-current process. Every operating superintendent has worked out series of power charts similar to Fig. 12 of Mr. Hansen's paper. The working part of this chart is that between 5 and 9 per cent. acid, and approximately 20 and 35 amp. per sq. ft. General practice has adopted 25 amp. per sq. ft., or slightly less, as the most economical current density. Working within these usual limits, a change of 15 per cent. in applied voltage with constant acidity will approximately double or halve the current density, whereas a change of 1 per cent. in the acidity of the solution, at constant voltage, will produce about a 15 per cent. change in the current density.

The problems of roasting, leaching, and purification having been largely solved, and a pure solution having arrived at the tank room, it is evident what ready means exist to determine what current density and rate of precipitation are most economical, power and other operating costs, interest on investment, and the price of zinc, all being taken into consideration.

As Mr. Hansen's Fig. 12 shows, there is no appreciable difference in the cathode production of the cell, per kilowatt-hour, at 25 amp. current density, between 6 and 8 per cent. acidity, and at 35 amp. the difference is not considerably greater. This change in acidity, however, which may be effected simply by altering the rate of flow of the incoming solution by means of the valves, will change the current density approximately 30 per cent. Of course, the actual acidity, which may be employed over long periods of time, is determined by other factors related to the leaching and filtration; but it is evident that "acid control" can be a most effective means of current control for the brief periods when necessary changes are being effected in the tank room, or some external condition alters abnormally the voltage available at the tank room. According to general experience, there is no occasion, 95 per cent. of the time, for anything but constant voltage in the tank room; and, for the remaining time, a means of varying the voltage 5 per cent. either way is all that is necessary.

### *Design and Selection of Electrical Equipment*

It is becoming apparent that all the conditions were not known or fully analyzed by the electrical equipment manufacturers who applied their machinery in the earlier electrolytic zinc plants. In these times of

high costs, thorough analyses should be made to determine exactly the requirements that the tank room must demand of the electrical equipment, analyses unbiased by what has been done before. In this connection, the writer frankly acknowledges that in his paper<sup>5</sup> "The Substation Problem of the Electro-chemical Plant," that part of the discussion dealing with the voltage range of electrolytic zinc plants was unduly influenced, unconsciously, by the known characteristics of the electrical equipment already installed in such service. I feel that there is opportunity for greater resourcefulness than has been evidenced in the past. Since the electrolytic zinc process is substantially a constant-voltage, constant-current process, like the aluminum reduction process, and, like it, a process which should operate 365 days out of the year, I am confident that plans will be made to employ the type of electrical equipment which has the lowest first cost and the highest efficiency over the longest operating period. Continuous efficiency will not be sacrificed and initial cost unduly increased simply to take care of occasional abnormal conditions which resourcefulness can meet in other ways.

I expect to see the elimination of the motor-generator set from consideration for such service, on account of its low operating efficiency and high first cost. I expect to see a larger number of simple, shunt-wound, rotary converters employed, the most efficient machine obtainable, such as are largely used in the aluminum reduction industry; and, where a small voltage variation is needed, this will be obtained simply by reactance in the alternating-current supply circuit and variations of the shunt-field strength. This "reactance and shunt-field control" method of obtaining small voltage variations has not been used largely in America, except in the aluminum reduction industry, and in some of the older street railway systems; but it has been used extensively elsewhere, especially in Great Britain and her colonies.

For electrolytic service really requiring voltage variations greater than 5 per cent. plus and minus, I expect to see the installation of the most effective type of booster rotary converter, after all the various factors in the tank room have been given full consideration. The reasons for confidence in the suitability of the booster rotary converter for such service are set forth at length in my paper to which I have just referred.

C. A. HANSEN.—Mr. Yardley seems to lay stress upon adapting the electrolytic plant to standard types of electrical apparatus. Personally, while I believe in keeping standard voltages, etc., in mind in laying out an electrolytic plant, I think that the electrical apparatus is a decidedly minor item in the plant as a whole.

A plant should be designed to do what it must do, and, in general, the electrical apparatus should be selected to fit in with the rest of the

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<sup>5</sup> *Transactions, American Electrochemical Society* (1917), **32**, 99.

design. Ordinarily, the substation equipment for an electrolytic zinc or copper plant will account for about 10 per cent. of the total plant cost, and it does not seem fair to let considerations of standard voltages, etc., in any way prejudice the design of the other 90 per cent.

Mr. Yardley's point that no great voltage range need be required of the direct-current generating apparatus is well taken. When working with zinc sulfate, no current flows through the cells until a voltage greater than 2.7 per cell is applied; the average voltage of the commercial zinc cell operating at full load is around 3.5. Obviously then, the whole range from zero load to full load may be taken care of by a voltage range between 2.7 and 3.5, or, say, by a 250 plus or minus 32 volts.

Such requirements can be very conveniently met with rotary converters and synchronous booster sets. With power at about \$25 per horsepower-year, there appears to be little hope of reducing the power cost per ton of zinc below \$14 for the electrolytic zinc plant.

The difference of 4 per cent. in efficiency in favor of rotary converters, as compared with motor-generators, is quite an important item (say \$25,000 per year for a plant averaging 100 tons zinc output per day). However, if a rotary converter is operated from a line of tricky voltage characteristics, or from a line subject to frequent lightning disturbances, this apparent saving is often wiped out in plant interruptions. The motor-generator equipment will unquestionably be less subject to these objectionable line disturbances.

It should also be remembered that the synchronous booster must provide the required voltage variation for the converter equipment, and that the booster set is a motor-generator with all of its efficiency limitations. Maximum efficiency for the synchronous converter-booster set is then to be arrived at by requiring minimum voltage range.

Perhaps a specific instance may be cited for illustrative purposes. One of the large electrolytic plants in the country specified a direct-current voltage range from 75 to 175, with a nominal load at the higher voltage approximating 4000 kw. To cover this requirement with a converter-booster set would mean a converter voltage of 125, and a booster range of 50. The required voltage range made motor-generator sets appear more favorable and they were installed. It actually developed that until 125 volts were impressed no current flowed through the cells; hence a converter supplying 150 volts, with a 25-volt booster, would have given more regulation than the plant required. In this particular instance, power costs approximately \$80 per horsepower-year, and the power conditions are very favorable for a converter installation. Had the converter equipment with small voltage range been installed, the power saving would approximate \$14,000 per year. However, the added power cost approximates only 0.035 c. per pound of product made, not an appreciable fraction of the production cost.

SIDNEY J. JENNINGS, New York, N. Y.—Mr. Hansen emphasizes the need of a perfectly pure zinc sulfate electrolyte for a satisfactory operation. The owners of the Tainton process assert that they are commercially operating a plant in England where, by employing high current densities, they are able to work upon an impure electrolyte and obtain a satisfactory zinc.

C. A. HANSEN.—Until a year ago, I think I was fairly familiar with all of the work being done both in this country and abroad on electrolytic zinc, and I am absolutely certain that there is no question as to the fundamental necessity for pure electrolyte. If I remember correctly, the Tainton process is characterized merely by its use of high current densities and high acidities. The behavior of the zinc cell under these conditions has been covered in the paper presented. The high current density renders one relatively independent of corrosion rates provided solution temperatures can be kept low, but the high current density militates against low solution temperatures.

## Electrostatic Precipitation

BY O. H. ESCHHOLZ,\* EAST PITTSBURGH, PA.

(Colorado Meeting, September, 1918)

THE electrostatic process of fume precipitation is an excellent example of the successful application of scientific knowledge to an industrial operation. Originally proposed for the precipitation of sulfuric acid mists, it has been extended until at present there is practically no fume-carrying gas to which the method cannot be applied. Among the more common applications are: the recovery of non-ferrous smelter flue-dust, iron blast-furnace dust, cement dust, and potash fume. It should be borne in mind that the process is restricted to the precipitation of suspended particles, either liquid or solid, and does not separate gaseous constituents. However, by proper temperature control, mixtures of vapors having different temperatures of condensation to either solids or liquids may be selectively precipitated.

The process consists essentially of subjecting suspended particles, including those too minute to be effectively acted upon by centrifugal or gravitational forces, to such an electrical force that they are driven from the gas stream, acting as a conveyer, and deposited upon a suitable receiving surface. This directional impulse is obtained from the interaction of charged particles and an electrostatic field of definite intensity gradient existing between two dissimilar electrodes, known as discharge and receiving electrodes. In principle, the equipment is equally simple. As shown diagrammatically in Fig. 1, it consists of:

- (1) A source of high-potential direct current, requiring:
  - (a) Source of alternating-current energy.
  - (b) Means of transforming from low voltage to high voltage.
  - (c) Means of converting alternating to direct current.
- (2) Precipitation chamber having:
  - (a) Transmission tubes for fume-laden gases.
  - (b) Suitably designed and disposed electrodes.
  - (c) Means for affecting removal of deposited solids.

Although Hohlfeld demonstrated, in 1824, that a smoke-laden atmosphere could be cleared by applying an electrostatic field, and while Sir

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\* Research Engineer, Westinghouse Elec. & Mfg. Co.



Oliver Lodge and A. O. Walker recognized the metallurgical possibilities of that phenomenon in 1886, every attempt to commercialize electrical precipitation failed, due to the lack of a suitable supply of high-voltage energy. As Dr. Cottrell says,

The first step toward practicability was of necessity a commercially feasible source of high-tension direct current. The obstacles to building ordinary direct-current generators lie chiefly in difficulties of insulation, and if this is avoided in individual machines by working a large number in series, the multiplication of adjustable and moving parts intrudes itself. On the other hand, high-potential alternating current technique has in late years been worked out most thoroughly and commercial apparatus up to 100,000 volts and over has been available for some years . . . . The voltage is thus transformed and then commutated at a high potential by means of a synchronously rotating contact maker into an intermittent direct current. This

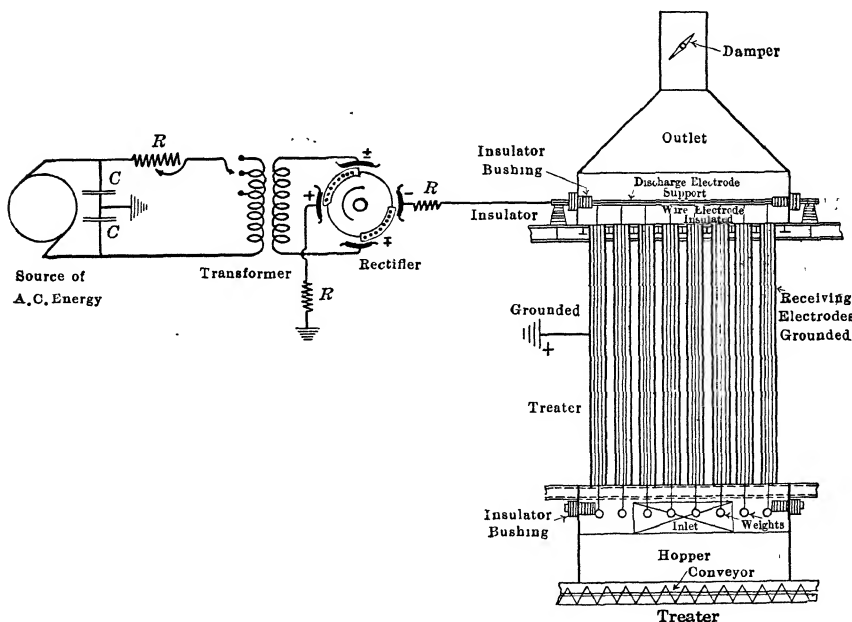


FIG. 1.—DIAGRAM OF TREATER CIRCUIT.

unidirectional current is then applied to a system of electrodes in the flues carrying the gases to be treated. . . . The development, therefore, may fairly be considered as the reduction to engineering practice, as regards equipment and construction, of the fundamental processes long since laid open by the pioneer work of Lodge, a feat vastly easier today than at the time of Lodge and Walker's original attempts.

In view of the important bearing that the development of electrical equipment has therefore had upon the commercializing of the precipitation process, it appears desirable to discuss briefly such apparatus, as well as some of the electrical phenomena characteristic of treater circuits.

## ELECTRICAL SYSTEM

Systems depending upon the use of a mechanical rectifier for the conversion of high-tension alternating current to direct current may be classified with respect to the source of power.

A. Low-tension alternating-current generator for treater load only:

- (1) Single-phase generator supplying power to single transformer-rectifier-treater unit.
- (2) Single-phase generator supplying power to two or more transformer-rectifier-treater sets operated in parallel on the low-tension side.
- (3) Multi-phase generator supplying energy to one or more transformer-rectifier-treater sets from each phase.

B. Low-tension alternating-current industrial or lighting mains having a relatively large capacity as compared with treater demand, and supplying one or more transformer-rectifier-treater sets.

C. High-tension alternating-current main supplying energy directly to potential regulator-rectifier-treater sets.

*System A-1*

Of the above systems, A-1 is most widely used, the consensus of opinion being that this arrangement insures: (1) Continuity of operation; (2) greatest flexibility; (3) maximum dust recovery; (4) utmost simplicity of control and operation.

This system has the very apparent advantage of definitely isolating the circuit for each treater from the main plant circuits, as well as from every other treater, so that all disturbances are localized. Since each generator serves but one transformer-rectifier-treater set, close and efficient control of treater voltage is obtained by simply varying the generator excitation. The flexibility of this system is perhaps its greatest asset, enabling the operator to meet the many exigencies arising from varying gas characteristics and to maintain a high rate of fume recovery

*System B*

System B has been applied to a relatively few installations in which the power requirement is large. Its chief merit lies in its low first cost. The equipment consists of a transformer-rectifier set (driven by synchronous motor) for each treater, with a series resistance in the primary circuit for securing control of treater voltage. Installations of this type have been usually viewed as temporary or experimental, and with increase in the units employed the following factors must be considered:

- (1) Effect of variations in supply-line on treater operation.

- (2) Effect of parallel transformer-rectifier-treater set operation, due to voltage wave distortion, on:
- (a) Performance of other industrial and lighting apparatus.
  - (b) Treater performance.
- (3) Hazard to life and equipment caused by reflection of high-voltage surges on line.
- (4) Limited control of treater voltage obtainable by varying primary series resistance.
- (5) Maintenance of heavy current carrying variable-resistance contacts.
- (6) Increased power loss due to this type of control.
- (7) Hazard of plant shut-down caused by treater disturbances.

### *Other Systems*

Recognition of the remaining systems has been urged at various times on account of their smaller first cost and floor space. A careful analysis of practically every project, however, has revealed that such saving is accompanied by a greatly increased liability of interruption to service.

In this effort to reduce the initial investment, it is overlooked that, while the commercialization of precipitating processes hinges upon the development of electrical units, the average first cost of the electrical equipment of representative installations is actually a small part of the total, approximating usually 10 per cent. and, in rare instances, 15 per cent. of the cost of the treater. Moreover, the value of the yearly recovery from the average installation is at least twice the total investment, ranging from 150 to 600 per cent. of the total first cost, depending upon the type of construction required and the character of the material precipitated. The cost of the electrical equipment is, therefore, roughly from 2 to 7 per cent. of the value of the yearly recovery. The tendency among engineers is therefore toward the development and installation of rugged apparatus, having a liberal margin, to assure continuity of service.

In addition to suitable generator, transformer, and rectifier units, it has been found desirable to include certain auxiliaries in the circuit, such as permanent series resistances in the high and low tension lines to dissipate oscillating discharges and limit short circuits; and condensers, shunted across the generator leads, to absorb infrequent high-voltage surges reflected from the transformer windings to the generator armature.

### TRANSFORMER

The extent to which the transformer incorporates the most advanced knowledge of high-voltage engineering is the measure of its success in precipitation service. Besides its obvious functions, it must bear the brunt of occasional surges caused by treater breakdown, and the periodic

oscillations produced at each half cycle on breaking the rectifier arc. Figs. 2 and 3 illustrate a type of construction in use in the majority of plants, which has demonstrated its ability to withstand, over a long period, the most severe operation encountered in precipitation service.

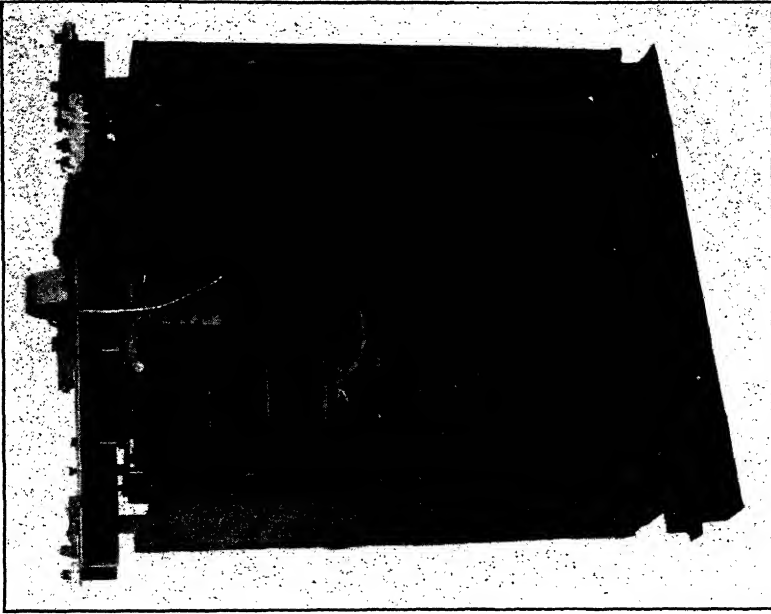


FIG. 3.—COILS OF 15 K. V. A. TRANSFORMER SHOWN IN FIG. 2.

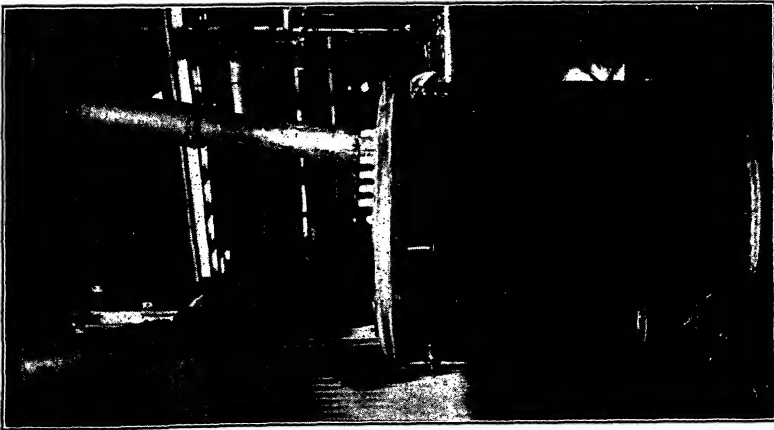


FIG. 2.—15 K. V. A. 100,000-VOLT, 60-CYCLE TRANSFORMER FOR PRECIPITATION SERVICE.

### RECTIFICATION

More dust is precipitated in a given time with a unidirectional field between electrodes than with an alternating field. With the latter, each reversal of field direction either retards the velocity of the charged

dust particles or completely reverses their direction, necessitating a larger and more expensive treater than is required with a field constant in direction. The problem of converting high-tension alternating current to high-tension unidirectional current has, therefore, received considerable attention. Although many devices, such as mercury rectifiers, hot-cathode converters, or kenotrons, high-voltage direct-current generators, unsymmetrical electrodes, etc., have been advocated, the mechanical rectifier is still in use on practically all commercial treater circuits. During more than 10 years, its peculiar fitness for this service

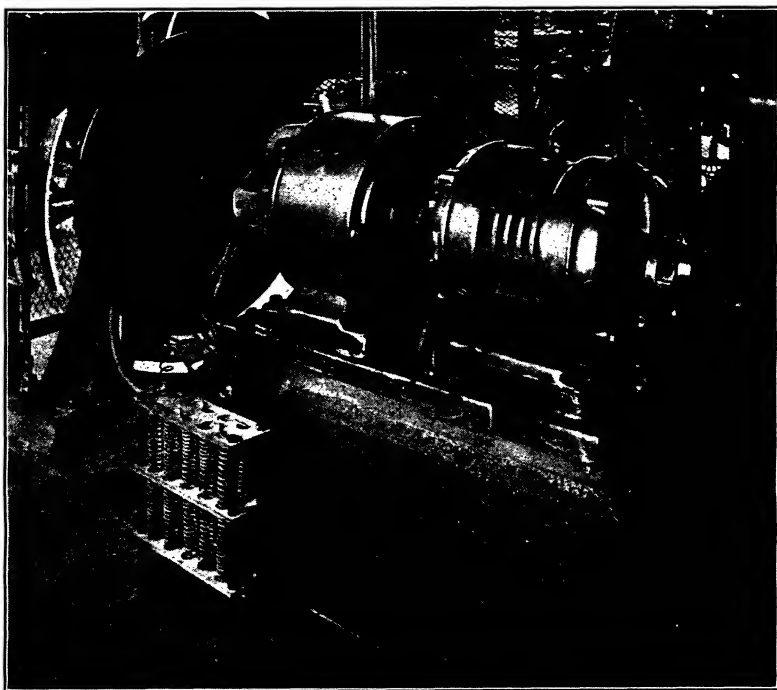


FIG. 4.—15 K. V. A. MOTOR-GENERATOR SET WITH DIRECT-CONNECTED DISK-TYPE RECTIFIER.

has been thoroughly demonstrated. As shown in Figs. 1 and 4, the rectifier consists of four stationary shoes, alternate ones being connected respectively to the transformer and the treater leads, and two conducting sectors mounted on the periphery of an insulating, rotating disk. During operation, the rotating sectors short-circuit adjacent and opposite shoes, so that at synchronous speed the alternating current is converted into direct-current impulses.

The noise accompanying the rapid breaking of the tail arcs as the rotating contacts recede from the collecting shoes is somewhat objection-

able. However, this is outweighed, in the estimation of operators, by such characteristics as:

- (1) Extreme simplicity in
  - (a) Operating principle.
  - (b) Mechanical and electrical construction.
- (2) Ease of adjustment to varying requirements.
- (3) Ruggedness, ease of repair and, therefore, low maintenance.
- (4) Low first cost.
- (5) Intermittent wave contact characteristic, which:
  - (a) Limits or suppresses treater short-circuits.
  - (b) Assists in sustaining treater voltage.

Despite the fact that the intermittent wave contact characteristic is well known as an inherent property of the rectifier, its effect on treater operation is not generally appreciated.

#### SUPPRESSION OF TREATER SHORT-CIRCUITS

Curve *A*, Fig. 5, illustrates a typical alternating-current or voltage wave obtained at the transformer high-tension terminals when operating on a resistance load without rectification. Curve *B* shows the wave form

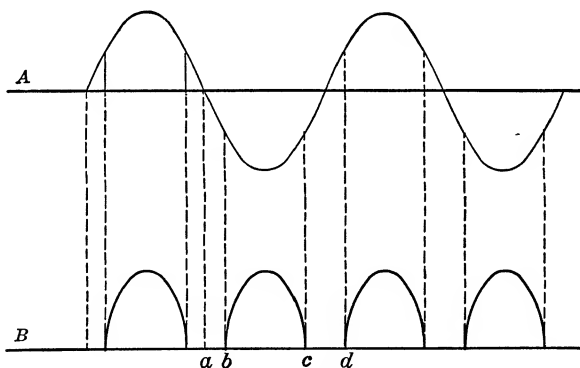


FIG. 5.—*A*. SINE-WAVE VOLTAGE OR CURRENT. *B*. UNIDIRECTIONAL VOLTAGE OR CURRENT WAVE OBTAINED ON RECTIFYING SINE WAVE *A* BY A MECHANICAL RECTIFIER WHEN SUPPLYING ENERGY TO A RESISTANCE LOAD.

modified by a mechanical rectifier. The period of wave contact *b-c* is determined by the angle subtended by shoes, rotating sectors, and tail arcs, while the interval *a-b* depends upon the length of gap between fixed and stationary conductors, and their position relative to the impressed wave when first making contact. Any section of the wave may be commutated by varying the length of rectifier shoes and adjusting them against or in the direction of the approaching wave front. Figs. 6 and 7 show, respectively, short and long periods of wave contact. Obviously, with the long contact, a greater amount of energy is transmitted.

Since a positive separation between the alternating and the unidirectional current circuits is secured during the interval *c-d*, a short-circuit occurring during the period of contact *b-c* will be immediately interrupted by the electrode separation obtained during *c-d*. As most treater short-circuits are local, being caused by changes in gas condition, irregular accumulation of deposits, or swinging of electrodes, this rectifier characteristic enables the treater to relieve itself by a heavy local discharge in a period usually less than  $\frac{1}{120}$  sec. with practically no interruption in operation or loss in recovery. It has been found possible, therefore, to operate Cottrell treaters for 24 hr. a day within 1 per cent. of the critical or breakdown voltage. Fig. 7 shows rectifier operation with the operating voltage maintained approximately 1 per cent. below critical voltage.

Fig. 8 shows the changes occurring in voltage (upper wave) and rectifier

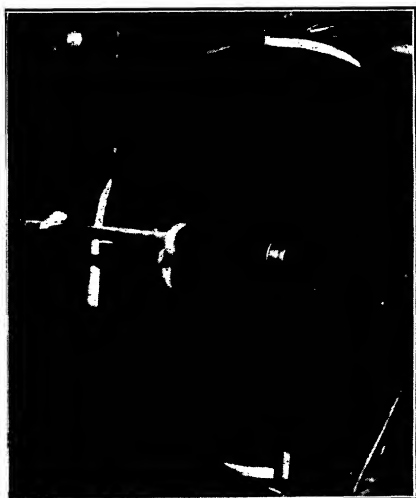


FIG. 6.—RECTIFIER ADJUSTED FOR SHORT-ARC CONTACT.



FIG. 7.—RECTIFIER ADJUSTED FOR LONG-ARC CONTACT.

ground current (lower wave) during the interruption of normal operation by a short-circuit in the treater. It will be noted that the short-circuit occurred near the peak of the voltage ripple, and cleared in less than  $\frac{1}{1000}$  sec., normal operation having been resumed in  $\frac{1}{120}$  sec. Only a slight increase in the rectifier current is evident at the instant of short-circuit. The current peak following the resumption of service is due to the capacity current absorbed by the partially discharged treater. With any other commercial method of obtaining high-voltage direct current, such an incipient short-circuit would have developed into a heavy current, imposing severe strains on connected apparatus, until the more or less complex control switches had cleared the line.

The rectifier cannot, of course, suppress a sustained short-circuit

caused by complete breakdown in the treater chambers, or by contact of electrodes. Under such conditions, it operates to limit the current until the circuit breakers open; this latter type of disturbance, however, occurs infrequently.

### TREATER VOLTAGE

As shown in Fig. 8<sup>1</sup> and particularly in Fig. 9,<sup>1</sup> the treater voltage is practically a flat wave. These oscillograms are typical of many that have been secured on treaters of different types and capacities. The inability of most investigators to record the treater-voltage characteristic is attributed to the use of an oscillograph of low sensitivity, or to excessive leakage of line, insulator, and treater current. The oscillograph, when connected as shown in Fig. 10, offers a discharge path for energy stored in the treater, and it is desirable, therefore, to use an

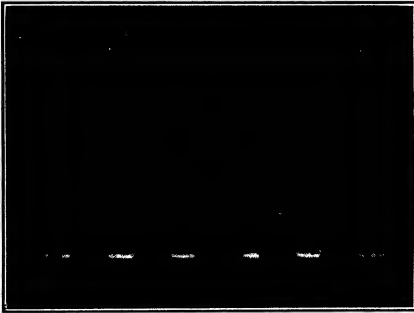


FIG. 8.

FIG. 8.—VOLTAGE ACROSS TREATER (UPPER WAVE) AND CURRENT THROUGH RECTIFIER GROUND LEAD (LOWER WAVE) IMMEDIATELY BEFORE AND AFTER SHORT-CIRCUIT IN TREATER CHAMBER. TIME INTERVAL BETWEEN WAVE PEAKS =  $\frac{1}{20}$  SEC.

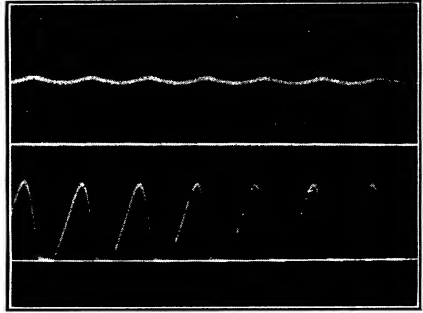


FIG. 9.

FIG. 9.—SHOWING SUSTAINED VOLTAGE ACROSS TREATER AND RECTIFIER GROUND CURRENT DURING NORMAL OPERATION. PEAK VOLTAGE 57,000; EFFECTIVE TREATER VOLTAGE APPROXIMATELY 97 PER CENT. OF PEAK. EFFECTIVE RECTIFIER CURRENT APPROXIMATELY 1 AMPERE.

instrument that will respond to the smallest changes of current; otherwise the fluctuations in treater voltage appear greater than actually occur during normal operation. In all of the treaters examined, the effective voltage ranged from 90 to 97 per cent. of the maximum. To check the oscillograph observations, comparisons of the actual effective and maximum voltages were secured by means of an electrostatic voltmeter and a sphere gap, the instruments having been calibrated together on a commercial sine-wave voltage. Fig. 11 shows the variation in effective voltage with a change in load at a maximum treater voltage of approximately 55,000. It may be noted that, throughout the operating range, the effective value is close to 95 per cent.

<sup>1</sup>These oscillograms were taken by H. I. Frisbie, Anaconda Copper Mining Co.



This sustaining of the treater voltage is caused by the comparatively slow rate of treater discharge. The suspended particles have usually an appreciable mass and, therefore, when charged under conditions where an arc is not formed, travel at low velocity. In fact, under the usual condition of moderate clearance of gas at a velocity of 6 ft. per second, obtained in a tube 12 in. diameter, 15 ft. long, the average velocity of the dust particles, at right angles to the gas stream is, roughly, but 0.8 in. per second, while complete precipitation of the same number of particles during the interval between breaking and making rectifier contact would require a velocity of 900 in. per second. Due to this slow rate of discharge, or ionic drift, the treater tends to maintain its voltage approximately constant, and the *rectifier practically operates to convert*

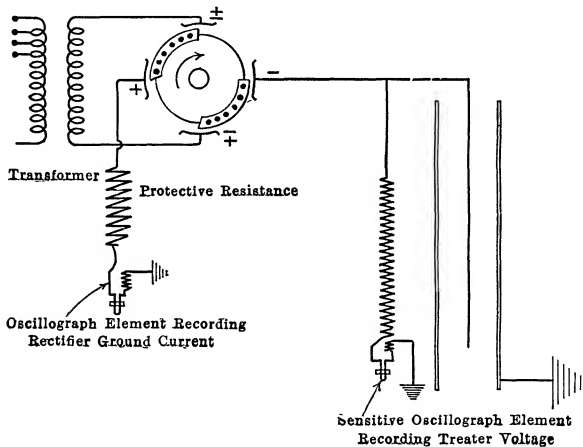


FIG. 10.—CONNECTIONS FOR OSCILLOGRAPH OBSERVATION.

a high-tension alternating current to a nearly constant high-tension direct current, so far as the flow across the gas space itself is concerned.

It is the author's opinion that a better utilization of the rectifier for suppressing short-circuits and sustaining treater voltage would result in improved clearance of dust, and increased returns on the investment.

#### OPERATING VOLTAGE

The treater voltage maintained with a given polarity of discharge electrodes is determined by such characteristics as velocity, temperature, conductivity, ease of ionization; also by electrode diameter, spacing, etc. In referring to the operating voltage, some confusion has resulted from the practice of assuming it as equivalent to the maximum effective voltage obtainable with a given transformer equipment. We hear repeatedly, therefore, of treaters operated at 100,000, 150,000 and even

250,000 volts, whereas very few commercial treaters are operating above 60,000 volts. The discrepancy between the assumed and the actual treater voltage is attributed to:

- (1) Use of a lower transformer ratio than the maximum.
- (2) Voltage drop across series resistances in high and low tension circuits.
- (3) Rectifier voltage drop.
- (4) Generator wave distortion from sine to flat-top wave by rectifier and treater load characteristics.

With further development in the control of gaseous ionization and agglomeration of suspended solids, the critical voltage will un-

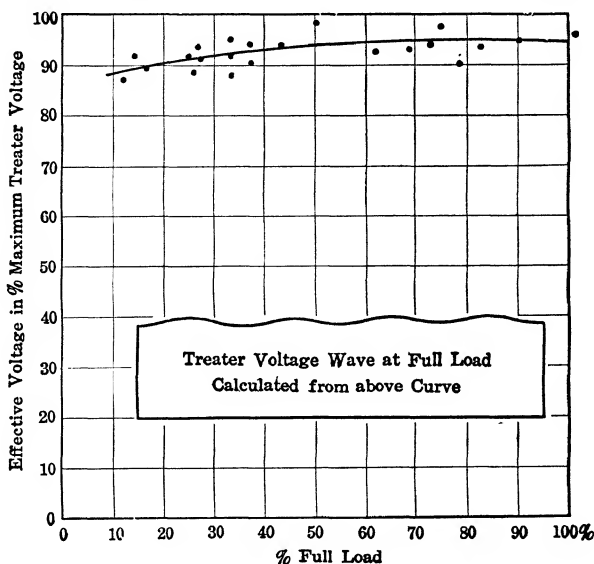


FIG. 11.—VARIATION IN TREATER EFFECTIVE VOLTAGE WITH CHANGE IN LOAD.

doubtedly be raised, and permit the use of a higher operating voltage with its attendant economies.

### ELECTRODE VIBRATION

One of the treater phenomena associated with high-voltage operation is the tendency of electrodes to vibrate. Non-rigid electrodes, of either the chain or the wire type, have been observed to vibrate in circular and elliptical orbits, whether supplied with direct current from hot-cathode converters, Girvin high-voltage direct-current generator, or mechanical rectifiers. The vibration may be intermittent or sustained. Either type, however, is produced by unbalanced electrostatic forces pulling the discharge electrode toward the receiving electrode, thereby increasing the field intensity until a local arc ruptures the inter-

vening gas. This attractive force increases as the square of the voltage. At 25,000 volts, it is quite negligible; at 50,000 volts, it may seriously interfere with treater operation; while at 100,000 volts, non-rigid electrodes become impracticable and are preferably displaced by rigid electrodes. Vibration occurring at 50,000 volts may be almost eliminated by careful alignment of discharge electrode, and by the use of connecting damping rods or chains to alter the natural vibration period of the electrode system, and prevent it from oscillating in tune with the periodic treater arcing or voltage pulses.

### TREATER PROBLEMS

In common with all radical developments, the electrostatic process is admittedly susceptible of further improvement. It is anticipated that valuable results will be obtained from the study of:

- (1) Variation of precipitation rate with:
  - (a) Change in gas velocity.
  - (b) Gas or vapor dilution.
  - (c) Accelerated agglomeration.
  - (d) Temperature change.
  - (e) Conductivity and ionizing potential of gases used.
  - (f) Conductivity, mass, dielectric constant of fume.
- (2) Methods for the removal of deposited material.
- (3) Control of gas distribution.
- (4) Control of the distribution and nature of deposits.

With regard to the last problem, Dr. E. R. Wolcott has shown that some deposits are essentially discontinuous dielectrics, particularly those obtained by the precipitation of cement dust, borax, etc., and form areas of high field density which reduce the critical and, therefore, the operating voltage from two-thirds to one-half of its initial value. The remedy now applied at a number of plants is to render the deposit conductive by subjecting the flue gases to a fine water spray. Where removal of the last traces of suspended fume is essential, as in the precipitation of potash, the absolute maintenance of a conductive deposit is assured by passing a continuous water film over the receiving electrodes. The potash treater recently installed at the Security Cement and Lime Co., Hagerstown, Md., embodies this development.

Investigations along the lines mentioned above will tend to:

- (1) Increase the percentage of recovery.
- (2) Decrease the cost of treater chambers, through increasing the rate of precipitation at high gas velocities.
- (3) Reduce operating cost and improve continuity of service through the development of automatic methods for the removal of deposits formed on either electrode.

## SUMMARY

(1) The commercialization of the electrostatic precipitation process has pivoted about the development of transformers.

(2) The synchronous contact maker, or mechanical rectifier, besides converting high-tension alternating current to high-tension direct current, serves to: (a) Suppress treater short-circuits, and (b) sustain treater voltage, thereby permitting operation within a few per cent. of the critical voltage of the treater, while also maintaining a practically constant voltage, resulting in high recovery of suspended substances.

(3) Rapid progress is being made in the control of disturbing phenomena, inevitable with high-voltage operation.

(4) The precipitator investment is approximately 8 to 10 times that of the electrical equipment. Improvement in construction of the precipitator will show, therefore, a correspondingly greater reduction in total cost of installation.

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## DISCUSSION

GERARD B. ROSENBLATT,\* Salt Lake City, Utah (written discussion†). —Mr. Eschholz attacks this problem from what appears to me to be the proper angle. He does not limit his viewpoint to the attainment of ideal results under conditions approximating laboratory practice, but rather discusses actual commercial conditions that must be maintained in large-scale operations, where continuity of service without expert attendance is one of the prime considerations.

I am of the opinion that Mr. Eschholz has not given sufficient consideration to the commercial possibilities of what he describes as System B, the use of low-tension a.-c. industrial or lighting circuits for supplying power to Cottrell treaters through the medium of a step-up transformer and a synchronously driven rectifier, without the use of motor-generators. It is true that, to date, few installations on a large scale have used this system, but I believe that when the conditions of power supply are suitable, and when proper precautions are taken, this system can be used successfully and to advantage where at present it is considered necessary to use motor-generator sets.

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† Received Aug. 19, 1918.

In the average commercial Cottrell installation of any size, the motor-generator set is used for two purposes:

- (1) To isolate the industrial supply circuit from the Cottrell circuit.
- (2) To afford an easily operated and smooth system of control for the Cottrell treater voltage.

With proper engineering, both of these objects can be accomplished without the use of motor-generator sets.

In isolating the industrial circuit from the low-tension Cottrell circuit, the motor-generator set performs two functions; it eliminates or at least damps the effect of irregularities of voltage in the industrial supply circuit upon the voltage of the Cottrell circuit; and it prevents disturbances in the Cottrell circuit from being reflected back to the industrial circuit.

If the voltage regulation of the industrial supply circuit is good, then the first function of the motor-generator set is unnecessary. Many central stations and many of our larger industrial plants, smelters, cement works, and the like, maintain voltage regulation which is altogether adequate for direct supply to the Cottrell transformer. Even if they do not, it may prove in many cases that a small automatic induction regulator of the type commonly used in lighting systems may remedy most of the deficiencies in voltage regulation. It is a matter of balancing dollars and cents to determine whether such a regulator is justified.

As to the reflection of Cottrell-circuit disturbances to the industrial circuit, this also can be guarded against by the proper precautions. The disturbances that are apt to emanate from the Cottrell circuit may generally be classified as follows:

- (1) The creation of disturbing harmonics by the parallel operation of synchronously driven mechanical rectifiers, which rectifiers cannot be set so that they make and break contact at exactly the same point in each cycle.

- (2) The effect of maintained short-circuits on the high-tension side of the step-up transformer, usually in the treater tubes themselves.

- (3) The reflection of high-voltage surges from the high-tension treater circuit to the low-tension side of the transformer.

The harmonics created by the parallel operation of mechanical rectifiers can be cared for in a number of ways. If the total capacity of the supply circuit is large in comparison with the energy taken by the Cottrell system, these harmonics are readily absorbed and cause no trouble whatever. This has been quite definitely brought out by certain oscillograph investigations which have been conducted with an installation of approximately 100 k.v.a. of Cottrell equipment in a western smelter. If, however, it is desirable to absorb these harmonics, it can probably be accomplished by one of several methods, none of which has yet received adequate investigation. One of these methods is to supply a

damping winding in the step-up Cottrell transformer. Another is the possibility of using a selective resistor which can be constructed to have very much higher impedance for abnormal frequencies than for the proper operating frequency. Another possibility is a synchronous impedance connected to and operated from the mechanical rectifier. Admittedly, all of these remedies for the absorption of harmonics introduce complications in the circuit, but with proper development it is very possible that these complications may be reduced to such an extent that, from an operating point of view, they will be negligible.

The effect of maintained short-circuits may be counteracted exactly as in the use of motor-generator sets; that is, by proper mechanical rectifier backed up by adequate circuit-breakers.

The reflection of high-voltage surges is also readily prevented by simple, permanently adjusted, immobile apparatus such as small condensers connected between line and ground, with possibly the addition of a safety spark gap. Condensers are now used for this purpose with most motor-generator installations.

With proper protective equipment, the life hazard mentioned by Mr. Eschholz is certainly no greater when using an industrial circuit supply for the Cottrell transformer than when using motor-generator sets.

The control of treater voltage when using an industrial circuit to supply the Cottrell transformer direct is probably not quite so accurate as when using motor-generator sets, but it is sufficiently fine for many commercial applications, and the advantages of eliminating the motor-generator sets, even at a sacrifice in fineness of control, should certainly receive consideration in many instances.

The maintenance of heavy current through variable resistance contacts is not so serious if the contacts are properly designed and if the voltage difference between adjacent contacts is kept low. It has been found that what burns contacts on the series rheostat, in the low-tension of a Cottrell circuit, is not the current carried by the contacts but the arc formed when the contact arm is moved from one contact to another. Admittedly, this means a rheostat having a considerable but not an inordinately great number of steps.

Under commercial operating conditions, I question whether the power losses would be very different in a system using industrial power supply than in one of individual motor-generator sets. The losses in the series adjusting resistance, and in the synchronous motor for driving the rectifier, probably would average about the same as the combined losses in the motor-generator set, including those entailed in the excitation of the Cottrell generator. Anyway, in a commercial plant, power required for electrical precipitation is a small portion of the expense of operating, and if one system did take 10 per cent. more power than another, it would be difficult to find its effect in the actual cost per ton of dust recovered.

The hazard of plant shut-down, which might be caused by a treater disturbance in case industrial power is used directly on the low-tension of the Cottrell transformer, is the one point that cannot be decided until some large commercial installation, omitting motor-generator sets, is actually made and operated for a considerable period of time. I think that, with proper consideration of the points previously mentioned, this hazard would not be serious.

The foregoing has been mainly confined to the disadvantages charged against the omission of motor-generator sets in a commercial Cottrell installation. The advantages of omitting the motor-generator sets are worthy of mention; they are:

- (1) Less space required for electrical equipment.
- (2) Less apparatus to buy and care for.
- (3) Smoother operation if the power supply has proper characteristics.

(1) Any tentative plant layout will prove the space economy of omitting the motor-generator sets, even considering the increased space required for the switchboard installation and admitting the fact that the rectifier, whether driven from a motor-generator set or a little synchronous motor, occupies a large proportion of the floor area required by all rotating machinery.

(2) The omission of the motor-generator set decreases the first cost of the apparatus but, as well pointed out by Mr. Eschholz, electrical apparatus is the small end of the first cost of the treater, and I would not lay too much stress on a slight reduction in first cost of electrical apparatus. The matter of maintenance and attention is of somewhat more importance, and I think that it will be found that maintenance of rheostat contacts and synchronous motor bearings will be less than that of motor-generator sets with collector rings and with exciters having commutators. However, the actual cost of maintenance, in dollars, may not differ greatly because an attendant is necessary in most Cottrell installations, and he usually has spare time to devote to keeping any *simple* design of machine in good shape. I believe, however, that shut-down due to machine trouble will be probably less if motor-generator sets are eliminated.

(3) The question of smooth operation if industrial power supply is used direct for the Cottrell transformers depends entirely on the characteristics of that supply. If the wave form is right, if the voltage regulation is good enough, and if the capacity behind the plant is sufficient, experience has shown that smooth operation in the Cottrell treater follows. If these conditions do not exist, then operation with industrial power supply will be very much more troublesome than if motor-generator sets are used. For this reason, the elimination of motor-generator sets from the installation for a Cottrell treater cannot always be properly



recommended; I contend merely that in many places industrial power could advantageously be used direct.

In any event, for a commercial installation, either the motor-generator set with mechanical rectifiers and step-up transformers, or industrial power supply direct to the step-up transformer with synchronously driven mechanical rectifiers, would seem far more satisfactory than any system which, in order to eliminate the mechanical rectifier, introduces intricate and as yet not thoroughly developed electrical equipment. If the Cottrell process is installed in a plant to make money from the product recovered, then the apparatus used must be of the simplest, most rugged, and most highly developed type.

HARMON F. FISHER,\* New York, N. Y.—Mr. Eschholz discusses the particular case of large precipitators installed in connection with large metallurgical operations, and receiving their high-potential energy through the conversion of alternating current to direct current by means of the mechanical rectifier. He classifies these mechanical rectifier installations, according to source of power, as follows:

*System A.*—Applications utilizing individual alternating-current generators, either single-phase or polyphase, installed exclusively for furnishing energy to one or more transformer-rectifier-precipitator units.

*System B.*—Applications having no special alternating-current generators, but utilizing the existing alternating-current industrial or lighting mains to supply the energy to one or more transformer-rectifier-precipitator units.

*System C.*—Applications receiving their energy from high-tension power circuits supplied directly to potential regulator-rectifier-precipitator units.

I do not intend here to enter upon a detailed technical discussion of the foregoing classes, which would really interest only the manufacturers of electrical apparatus and the electrical engineers. A brief general discussion follows:

#### *System A*

Remembering that the paper under discussion is confined entirely to precipitation installations as applied to large metallurgical plants, the author is undoubtedly correct in stating that system A-1 is at present the most widely used in these applications. In this case, a generator is installed exclusively for the purpose of furnishing energy to one transformer-rectifier-precipitator set, thus permitting the greatest flexibility in operating control. By reason of this flexibility, it may be possible, in a few instances, to improve slightly on the dust recovery attainable with the simpler installations listed under system B, but as a general rule, the simpler installations can be adjusted to give any degree of clearance

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desired. A system utilizing individual generators does not, in our opinion, afford any greater continuity of operation than is possible with the simpler installations.

### *System B*

It is our opinion that, of the three systems outlined in the original paper, the simplest arrangement is that of system B.

Considering precipitation as applied to all industries, undoubtedly this is and will remain the most widely applied system. It is recognized that it should not be applied to an existing power main having an energy capacity but slightly in excess of the precipitation demands, nor on a power main subject to violent fluctuations.

The author states that voltage control is obtained by means of a series resistance in the primary circuit of the transformer. There appears to be some confusion on this point; voltage control is intended to be obtained by transformer primary taps. A certain amount of voltage control can be obtained by varying the resistance in the transformer primary, but a distinctly separate and valuable property of this resistance must not be overlooked; namely, it helps to absorb the energy of the surges and oscillations which the rectifier-precipitator load impresses upon the transformer circuit.

Most modern industrial lighting and power circuits have fairly close voltage regulation; hence, in only abnormal cases will there be severe variations in the supply line which may prove detrimental to precipitator operation.

The effect of multiple-parallel transformer-rectifier-precipitator operation on voltage distortion will generally be more pronounced in system A than in system B. The effect on other industrial and lighting apparatus on the same circuit, the hazard to life and equipment caused by reflection of high voltage surges on the line, etc., are rather remote possibilities, particularly if the same auxiliary protection is installed that Mr. Eschholz mentions in the final paragraph under "Other Systems."

It may be mentioned that cases are on record where, on account of faulty operation, individual generator-transformer-rectifier sets have been replaced by synchronous motor-driven rectifier-transformer sets operating directly off the main low-potential power supply line, with greatly improved operation.

### *System C*

This system, utilizing energy directly from a high-potential alternating-current main, has never been seriously applied, to our knowledge. Besides having no advantages and being very awkward to operate, it would have many disadvantages over the two systems already discussed.

*Rectification and Electrode Vibration*

As stated in the original paper, one of the sturdiest and most reliable pieces of apparatus in use to-day is the modern high-tension transformer. Under even the most severe and violent operating conditions of the past, before our present knowledge of electric circuit conditions and gas conditioning were available, this apparatus gave but very little trouble and a very nearly perfect record may be expected in the future.

For converting alternating current to direct current, the old mechanical rectifier, because of its extreme simplicity, ruggedness and ease of repair, will always appeal to the practical operating man who has become accustomed to its use.

An important property of rectifiers, because of the rapid alternate opening and closing of the high-tension circuit, is the prevention of long continuous arcing or surging in the precipitator, due to any temporary upset of the electrical equilibrium of the circuit. This feature of rectifiers is also useful in instances where the electrode spacing has become less than would correspond to the potential at which the precipitator is operating, or in the case of an accidental short-circuit. Here it prevents abnormal current rushes, and serves as a limit to the short-circuit current to which a given transformer equipment would otherwise be subjected.

The author mentions electrode vibration as being due to unbalanced electrostatic forces. These same forces have been found to exist in the most carefully conducted corona experiments, where practically perfect centering of the electrodes within the outer cylinders could be assured. The remedy is relatively simple, and consists in placing damping rods, pipes, or chains in the electrodes so as to interrupt the simple harmonic vibratory motion. Chain electrodes are less subject to this phenomenon than a single wire electrode, and in no instances has any serious trouble been experienced from this cause.

Under "Treater Problems," the author mentions certain lines of studies for the improvement of the precipitation art. It may be of interest to know that the more important of these have been carried on for an extended period, and are being conducted at the present time, and results have been obtained which indicate the possibility of decided improvement both in the size of the precipitators and the smoothness and continuity of operation.

B. L. SACKETT,\* Tooele, Utah.—It may be interesting to record some actual conclusions derived from two years' operation of Cottrell precipitators at the Tooele, Utah, plant of The International Smelting Co. In emphasizing the importance of installing the best of electrical equipment in Cottrell power-houses, Mr. Eschholz states a fact which

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\* Blast-furnace Superintendent, International Smelting Co.

cannot be disputed, for if the electrical equipment be inefficient or insufficient, the effect is at once noted in unsatisfactory treater recoveries. However, it does not necessarily follow that the most costly installation gives the most efficient results, as the experience we have had at one of our treaters has shown.

There are two treaters at the Tooele plant which have been in operation for about two years; a four-unit, 880-pipe treater working on the gas from the Dwight-Lloyd sintering plant; and a one-unit, 220-pipe treater, working on the gas from the copper converters. In both of these treaters, pipes of 12 in. diameter and 15 ft. long are used. An additional four-unit, flue-type treater, which will treat the gas from the McDougall roasting plant, is now in course of construction.

The original electrical systems installed at the first two of the above mentioned treaters were: at the sintering-plant treater, a single-phase generator supplying power to four transformer-rectifier-treater sets operated in parallel on the low-tension side. (Mr. Eschholz's classification A-2); at the converter treater, a single-phase generator supplying power to a single transformer-rectifier-treater unit (Mr. Eschholz's classification A-1.)

Our work at Tooele during the past eight months has led us to conclude that improved treater operations can be obtained by using the electrical equipment classified by Mr. Eschholz as System B, namely, low-tension, alternating current from the smelter industrial main, supplying power to the transformer-rectifier-treater sets, rather than system A-2 or A-1.

The four-unit sintering-plant treater has been operating on system B for over two months, giving noticeably improved treater efficiency as compared with that obtained with system A-2, due to an increased power input to the treater, and a smoother wave form, as shown by oscillograms. The one-unit converter treater will be continuously operated by system B as soon as the necessary equipment arrives. The new four-unit McDougall roaster treater will also be electrically equipped by system B.

In all of our work at these treaters, both experimental and operating, it has been shown to our entire satisfaction that the undesirable possibilities to be looked for when using system B, as given by Mr. Eschholz, really do not exist if the installation be properly made. It is actually found that system B gives smoother electrical operation, higher power-plant input, and consequently, improved treater efficiency, than did either of the other installations; and at no time has there been the slightest indication of any reflected disturbances on our main power line from either treater.

Mr. Eschholz states that the first cost of the electrical equipment for a Cottrell treater is approximately 10 per cent. of the total cost of the treater. In the case of our sintering-plant treater, the cost of the

electrical equipment was 10.2 per cent. of the total cost of the treater; at the converter treater it was 15.3 per cent.

In connection with the cost of electrical equipment, we have found that the repair charges for this equipment have mounted up to a figure that is worthy of serious consideration. For example, at our sintering-plant treater, repairs have cost slightly over 40 per cent. of the first cost of the equipment in less than two years' operation.

Mr. Eschholz also states some general figures as to the value of the yearly recovery from the average Cottrell installation. The annual net profit derived from our treaters, to be applied to construction cost, is much less than that indicated by Mr. Eschholz as a minimum. In speaking of this profit, the figures given represent the gross value of the metals contained in the recovered fume, less the treater operating costs, including repairs, the cost of smelting the fume, and making proper allowance for the metallurgical losses in the smelting operation. The periods of operation represented are 22 months, and 19 months, respectively, for the sintering-plant and converter-plant treaters. The calculations are based upon the prevailing metal prices for the respective periods under consideration. At the sintering-plant treater, these profits have amounted to approximately 38 per cent. per annum of the total first cost of the treater. At the converter treater they have been about 56 per cent. of the first cost per annum.

Had the average metal prices for the past 10 years been used as a basis for these calculations, other factors remaining as they actually were, the sintering-plant treater would have shown a slight loss instead of profit, and the profit per annum at the converter treater would have been slightly less than 8 per cent.

L. D. RICKETTS, New York, N. Y.—May I ask what is the purpose of the Cottrell treater in the converter plant?

B. L. SACKETT.—The prime reason for both treaters was to abate the smoke, the familiar question of smoke litigation having arisen, which we expected to avoid by removing the solids from our escaping gases.

L. D. RICKETTS.—After you began using the Cottrell treaters, and recovering your lead in the form of sulfate, did you then begin converting the lead matte from your blast furnaces?

B. L. SACKETT.—No; we do not handle the fume from our lead converters in the Cottrell treaters. That goes to the bag house, being a product which is completely neutralized. Our copper matte carries some lead, which we were anxious to recover, as well as to abate the smoke damage. It is only the copper-converter dust that we are catching in the treater, while the gas from the lead converter goes to the bag house.

H. D. RANDALL, \* Salt Lake City, Utah.—One important point, from the electrical standpoint, is the fact that the small machine of system A is apt to have a poor wave form as compared with the large machine in a central station. Also the limited electrostatic capacity of System A easily sets up harmonic vibrations of the higher frequencies. System B is electrically a part of the transmission system feeding it, and owing to its comparatively enormous electrostatic capacity, is not subject to the phenomenon of resonance. The comparison is analogous to a violin string against an aerial tram cable.

This, in itself, is the strongest argument in favor of system B; and now that central stations and transmission lines have such perfect voltage and frequency regulation, there really is an extremely small advantage, if any, to be attained from separate motor-generator sets.

E. P. MATHEWSON, New York, N. Y.—There are certain practical points connected with the operation of the Cottrell plants which I think should be discussed, although they are not directly brought up by the author of the paper.

In the early days of the Cottrell apparatus, they treated moist fumes or gases carrying material that could be precipitated—sulfuric acid was the first. The treating of dry gases seemed to be a problem. Lately, we have been gathering information from various plants which indicates that a certain amount of moisture in the gases is necessary for good precipitation of the dust. At one plant, a relative humidification of 42 per cent. is considered the best proportion. This has been obtained by sprays of water from the roof of the flue leading to the Cottrell apparatus, and since those sprays were put in, the results have shown a wonderful improvement. At the plant of the International Smelting Co. at Miami, the concentrates treated in the roasters to which the apparatus is applied contain so much moisture that the gases naturally have the proper proportion, and the results at that plant are extremely good.

When visiting a plant in the East not long ago, I noticed that the motor-generator set originally installed had been discarded, and a small synchronous motor had been put in to drive the rectifier. The superintendent explained that they took their power from a public service company, and had no trouble whatever from surges on the line, and no complaints at all from the power company since installing this synchronous motor, but that they had experienced no end of trouble in operating their Cottrell treater so long as they used a motor-generator set.

I called the attention of some of the superintendents at our various plants, which were using Cottrell treaters, to this fact, but it only seemed to touch a sore spot with them; it seemed that there had been a great

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\* Manager, General Electric Co.

deal of discussion among the superintendents of the various plants, as well as the electrical experts, on this point. Some of those who had been instrumental in designing installations thought that the dangers from surges were so great that the use of synchronous motors was absolutely unsafe, and that the possible saving of a few dollars should not be considered at all in the installation of the Cottrell treater; that the main thing was to get the dust precipitated, and that a slight increase of efficiency in the electrical end of the apparatus was not worthy of consideration.

On my present trip, I have found that the electrical expert in charge of the precipitation department in one of the plants has become fully convinced that the synchronous motor is the proper apparatus to install, and he demonstrated this to my untechnical eyes very clearly. They had four motor-generator sets on each side of the center of the building, with transformers corresponding to each set. Four of these motor-generator sets had been re-wound, so that they were practically synchronous motors, and were driving the rectifiers. The power was supplied by the Montana Power Co., a large source of power. I could see plainly the sparking above the transformers of the motor-generator sets, while there was no sparking whatever on the other side of the building where the synchronous motors were used. The voltage on the side where the sparking occurred was 25,000, and on the other side was 27,000 to 28,000. It proved to me that it would be wise for all who are contemplating putting in the Cottrell apparatus to consider carefully the difference in these two methods of applying the power to the rectifier.

There is one substance which non-ferrous metallurgists have always found to give a great deal of trouble in precipitating—that is zinc oxide. Numerous experiments have shown that the Cottrell apparatus is not adapted to the precipitation of zinc oxide where it occurs in large quantity. The zinc oxide, apparently—I now speak in a non-technical way—is precipitated in such form as to make a non-conductor, and the precipitation continues only a short time before its efficiency is greatly impaired. To save this substance it seems to be necessary to use some filtering agent, such as the bag house.

F. H. VIETS,\* New York City.—The Research Corporation has done a great deal of work on the zinc oxide problem, and has built a treater at a brass company's plant in Connecticut to recover the zinc oxides from their casting shops. The principal factor in treating that fume is its humidification, which requires close adjustment. The last time I visited Torrington, last September, they were making about 85 per cent. recovery with this experimental unit.

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\* Engineer with Research Corporation.

In comparing the two systems of supplying power to precipitator circuits, a certain technical point requires explanation: System B, in which a large source of power supplies current of low voltage to precipitator circuits, is not nearly so liable to oscillations as is a small generator with its armature immediately adjacent to the primary of the transformer, which may act as a mirror to reflect back to the transformer any oscillations that may occur there. These oscillations may build up dangerous potentials on the secondary or high-voltage terminals of the transformer. The larger power system does not lend itself so readily to these oscillations; at least, it does not permit them to grow to dangerous proportions. System B has recently been improved so as to be controlled by a potential regulator on the primary, or low-voltage circuit, which gives very close regulation, and very efficient operation between the taps on the primary of the Cottrell transformers. These transformers are provided with taps on their primaries so as to give approximate voltage regulation. Between these taps, until recently, it has been the custom to use a series resistance to obtain voltage regulation.

I might comment briefly regarding recent observations on the rate of dust migration; I have in mind the case of fume-laden gas escaping from a 50-ton commercial kiln calcining alunite ore for the production of sulfate of potash. This gas contains not only very finely divided fume, but also some alum and coarse dust. When treating this gas in a pipe of 12 in. diameter by 15 ft. long, we find by filtration tests that over 99 per cent. of the solids entering the pipe are precipitated when a gas velocity of 7.5 ft. per sec. through the pipe is maintained. This means that the fume particles themselves are in the pipe for 2 sec. Considering the comparatively small number of particles entering near the center of the pipe, and assuming that they do not reach the pipe wall until they travel the whole length of the pipe, the particles must travel the 6-in. radius in 2 sec., and must have a velocity, due to electrical stresses, of 3 in. per sec. As a matter of fact, most of the dust is precipitated in the lower 6 ft. of the pipe, the upper 6 ft. recovering relatively small quantities of the dust.

B. L. SACKETT.—Regarding humidification, we have found, in the case of our converter treater, that it is absolutely essential to add a considerable quantity of water in the form of a fine mist; before adding that moisture we were not able to maintain anything approaching the proper voltage in the treater, and our recoveries were extremely low.

The gas going to the treater enters at a temperature, before being sprayed, of approximately 300° F. (150° C.); while the sprays do not lower the temperature very much, they seem to add sufficient moisture to accomplish the result required.

We have also recently learned that the Western Precipitation Co., of



Los Angeles, has found that gases which have been rather ineffectively handled heretofore are now successfully treated with the aid of an efficient spraying system.

F. G. COTTRELL,\* Washington, D. C.—It is interesting to notice that points raised tonight, as still under active discussion, were the centers of interest in a great deal of the work in the first few plants erected.

The use of the synchronous motor for the rectifier drive was naturally the first method adopted in the laboratory, and was carried into the initial commercial installations at Selby, and later at the Balaklala plant, although even at Selby, as development progressed, we tried out separate generators. At that time small a.-c. generators were not so easily obtainable as today. The development of wireless and other demands for small a.-c. generators have since brought the technique to the point where it is today, but at that time we had to content ourselves with a made-over four-pole d.-c. motor, having a special armature wound for it; but armature reaction in that case was very high. This, run as an a.-c. generator, would carry a lamp-load very nicely, but we could not get more than a few per cent. of its rating out of it on the rectifier load.

In the Balaklala plant we went over to the three-phase combination at the start, but found that, while this permitted us to get a little more precipitation out of each treater, the power consumption was greater in proportion than the gain in efficiency in the treater, and so we went back again to the single-phase.

After that, gradually, the commercial availability of independent small generator units came about, and they were adopted in some of the later plants, but that was mostly after I ceased to be in close contact with the work, and my knowledge of results is, therefore, largely second-hand.

Even the matter of zinc oxide came prominently to our attention at Balaklala. With the high-zinc ores which they smelted at certain times, we had difficulty with bone-dry precipitation, finding it hard to hold the voltage. We went into the question of humidification and rigged up sprays which we found helped us out. When the total zinc in the ore was low, the acid in the roaster fumes was sufficient to make the deposit slightly conductive, but with high-zinc the oxide was sometimes in excess, and made trouble. We were inclined to the conclusion, at that time, that it would be necessary to have a slightly conducting precipitate, but our ideas as to just what was necessary were so often reversed in that early work that I became very cautious of drawing any general conclusions.

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\* Chief Metallurgist, U. S. Bureau of Mines, and inventor of the process here under discussion.

That was particularly true in the application of the process to cement works, where very high temperatures were to be encountered and there could be no appreciable condensation of acid or moisture. But Mr. Schmidt, at Riverside, was soon running his precipitators up to temperatures of  $450^{\circ}\text{C}$ ., and even higher, with cement dust, which was about as dry and non-conductive, in the ordinary sense, as anything one could well imagine; yet he had no difficulty from these static troubles such as we had encountered with dry deposits at Balaklala.

We therefore had to revise to some extent our ideas of the significance of the non-conductive character of the fume. That still stands out as a rather interesting difference between cement dust, for example, and zinc oxide, at the same temperatures, one giving very much more trouble than the other. The essential difference in the electrical behavior of the two deposits seems most likely to be sought primarily in the fineness of their textures.

G. B. ROSENBLATT.—The choice between motor-generator sets and synchronous motors for driving the rectifier involves a point very often overlooked, which is the wave form of the power supplied to the rectifier itself.

The Anaconda Copper Mining Co. is installing the largest Cottrell treater in the world. They will treat dust from their copper roasting plant, and will employ a total of thirteen 75-k.v.a. outfits. After a rather lengthy investigation, with many trial runs on a more or less commercial design of treater, employing about 75 k.v.a., they decided on motor-generator sets. Personally I am not holding a brief for the motor-generator, but am merely transmitting to you information given to me by Mr. Murphy, of the Anaconda company.

Their investigations carefully covered systems A-1, and B, of Mr. Eschholz's paper. They gave no consideration to system A-2, because of the conjectured possibilities of regulation troubles. Their cost estimates led them to believe that the investment for system B, using induction regulators, as mentioned by Mr. Viets, would come to so little less than the investment for system A-1, that their appropriation was finally approved on the basis of system A-1, and the plant will be erected on that basis. They anticipate better operating conditions, less liability of interruption, and more general satisfaction by using motor-generator sets than by using the power of the Montana Power Co. direct on synchronous motor-driven rectifiers. They are, however, putting in one unit according to system B, so that they may have some actual comparisons under commercial operating conditions.

In connection with their tests under commercial conditions, the Anaconda company made a great many oscillogram tests, the study of which led to the conclusion that if a motor-generator set could be obtained

that would give as perfect a wave form as the average commercial power circuit, then the motor-generator set would afford superior operating conditions. If, on the other hand, the motor-generator set had shortcomings in its wave form, and embodied certain harmonics, then trouble might be looked for, not because of the motor-generator set itself, but because the wave form was not of the best. The smoothest operation may be expected with sets giving the most perfect wave form—more perfect than that of the average industrial power supply.

Anaconda has probably done as much as anyone toward developing the mechanical rectifier. They carefully examined the possibilities of other forms of electrical apparatus for obtaining uni-directional current, and considered everything, I believe, that could be furnished by the manufacturers. They gave particular attention to the hot cathode converter, or "kenotron," as it is called by its principal manufacturer. After testing it, and realizing its inherent disadvantages, they returned to the mechanically driven rectifier, and have developed it to a very high degree. They are now making and using the largest mechanically driven rectifier in the country; it is 42 in. in diameter, the largest previously developed being 36 or 37 in. They say they are getting excellent results from these 42-in. rectifiers.

E. E. THUM,\* Salt Lake City, Utah.—In regard to some of the practical points on moisture content of fume and on precipitator construction brought out by Mr. Mathewson, it might be interesting to describe a plant about to be built at the Southwestern Portland Cement Co. at Victorville, Cal.

Dr. Cottrell spoke of the work of Mr. Walter Schmidt and his associates of the Western Precipitation Co. on hot cement dust. The Riverside plant, installed under their direction, is a very fine plant for the precipitation of hot dust. However, I was told by some of their men that while the precipitation made a fine recovery of dust from the furnace gases, it allowed a considerable quantity of the finest fume to get by. Unfortunately, the fume is richest in potash, and as you all know, cement-dust potash is an important and valuable commodity at the present time.

The Victorville people investigated different methods for recovering the potash volatilized from their cement kilns, and Mr. J. G. Dean, their chemical engineer, decided that he would install a humidification process rather than the electrical process of precipitation. Mr. Dean's experimental plant takes the hot gases, sprays them carefully and thoroughly, thus cooling them to about 110° C. by contact with cold water and by the evaporation of the atomized spray. These gases now

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\* Western Editor, *Chemical and Metallurgical Engineering*.

contain a large percentage of water vapor and are passed through a condenser, there acting as a heating substance.

The condenser itself is rather unusual, the outside and supporting structure being made of reinforced concrete; the tubes and flue sheets are of steel, however. Within the condenser proper, a 24-in. vacuum is maintained above the weak solutions to be evaporated. Thus the humid gases passing through the tubes may be cooled to about 60° C. before discharge. Meanwhile the contained water vapor, condensing at 100° C. and below, would condense about the very small particles of potash as nuclei. The mist formed largely collects on the inside of the condenser tubes and trickles down into a sump, naturally carrying the potash in solution.

This works very nicely, but it is hard to collect mechanically the last particles of fog, and Mr. Dean found that even after the gases had passed through the condenser and been cooled and clarified, they still contained a considerable amount of unwetted potash particles. The amount varied somewhat—the potash recovery being perfect at times—yet under certain undetermined conditions of burning and wetting, the loss was large. Thus he finally decided that his humidification process was not as good as it might be, although it is producing about a ton of potassium sulfate a day.

Therefore he is now designing a Cottrell plant for treating these very cool, saturated gases which come through from the spray chambers and condensers, which is about as different from the gas at Riverside as one could imagine. Preliminary experiments show that the effluent from a precipitator treating these gases is sensibly potash-free at all times. The treater will contain concrete instead of iron pipes for the grounded electrode. Since the plant will precipitate a mist out of a saturated gas which is constantly cooling, the tubes will be always wet from this potash solution flowing down on the inside. This conducting film obviates the necessity of providing a metallic conductor for the grounded side of the apparatus. Since the gases are saturated on entering and cool during their passage, no evaporation of this film is possible, so no break in the circuit need be feared. Encrusting salts and growth of crystals are also impossible in the absence of any evaporation.

B. L. SACKETT.—Mr. Thum brings out a very interesting point and one that we have found important. He speaks of the ease with which the flue-dust, or the coarser particles of it, may be caught, and the difficulty of catching the finer particles.

As I understand it, the potash, as it occurs at the cement mills, is in the form of a fume; that is, has been formed by a chemical action, and is not a mechanical dust. At our plant we have found that the difficulties of precipitating a fume of this nature are far greater than with a straight flue-dust.

L. D. RICKETTS.—This point is well taken. In the treatment of converter gas at Miami, according to careful tests, we have very little copper escaping. The gas is hot, the solid particles contain a high percentage of copper and are practically all precipitated, yet the fume is dense white and practically all of the lead and zinc escapes. I wish to call attention, however, to the fact that the cooling of gases by admission of steam may be done satisfactorily so long as there is any zinc or lead oxide in the fume, but unless this is the case, free sulfuric acid will unite with the moisture to form a dilute solution which will destroy any iron work with which it comes in contact. At the Miami smelter we had to close down on account of a strike. It is probable that our steel stack discharging the reverberatory fumes had an internal coating of dust which was pasty with concentrated sulfuric acid, and that when the close-down came this acid absorbed moisture. In any event, it practically destroyed a portion of the stack.

E. R. WOLCOTT,\* Los Angeles, Cal. (written discussion†).—When the deposit of dust or fume produced by electrostatic precipitation is non-conducting, the electrical charge is retained by the deposited particles. If the dielectric constant of these particles be large, the accumulated charge may attain a considerable potential. In extreme cases this has been found to be of sufficient magnitude to produce ionization of the gases surrounding the deposits, an effect which is of course detrimental to satisfactory precipitation of the dust and fume. This “back ionization” has been photographed and reprints will appear shortly in the *Physical Review*. “Back ionization” is recognized by the fact that the voltage that can be maintained is lowered, sometimes as much as 50 per cent. of that which can be maintained with clean electrodes. This may be explained by considering that the “back ionization” increases the conductivity of the gases immediately surrounding the deposit, which is equivalent to moving the collecting electrode nearer to the discharge electrode, thus requiring less voltage to produce an arc.

Any means for making the deposits conducting, such as the addition of water, prevents this “lowering of the arcing voltage.”

LINN BRADLEY,‡ New York, N. Y. (written discussion§).—In reading Mr. Eschholz’ paper on certain types of electrical equipment for the Cottrell process, it might be valuable to consider just what real differences there are between a separate motor-generator set and a synchronous-motor set. When it is seen that the difference is slight, the question arises as to whether the results may not depend upon other features than

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\* Western Precipitation Co.

† Received Sept. 1, 1918.

‡ Chief Engineer, and Acting Manager, Research Corporation.

§ Received Sept. 14, 1918.

those brought out in the paper. In both types the precipitators are the same, the transformers and rectifiers are the same and, for good practice, the non-inductive resistance should be approximately the same.

The main differences are that in one case the alternating-current generator is in the precipitator power-house and the rectifier is driven by means of a mechanical coupling, while in the other case the alternating-current generator is in some remote power-house and the rectifier, for convenience, is driven by a synchronous motor which is merely a substitute for the mechanical coupling. In the former case, the voltage impressed upon the transformer may be varied both by action upon the generator field and by non-inductive resistance between the alternator and the transformer. In the latter case, voltage regulation is limited to one method, except for transformer taps, in usual practice, but experience has demonstrated that such regulation is indeed very effective and should be employed to some extent in either case.

Now if there is but slight difference in the regulating means, the difference in the size and characteristics of the alternators should receive more attention. It would seem that the large-sized and well designed alternator would be preferable as a source of the alternating current, and direct comparisons confirm this assumption. Of course, in order to get good results, the voltage delivered to the transformer should be steady, and if local conditions do not give steady voltage, due to intermittent operation of large motors on inadequate power circuits, it is necessary to correct the fault or else resort to motor-generator sets.

The facts that the precipitator load is not continuous, and that the load current is unusual, due to the laws of corona discharge, should be considered in designing a generator, since the rate of current flow at peak voltage is not the same as when other loads are employed. This accounts for the poor wave shape from an undersized or poorly designed alternator.

R. B. RATHBUN,\* Salt Lake City, Utah (written discussion†).—While the engineer should carefully weigh the merits of the various types of equipment, he must bear in mind that the object of his plant is the recovery of suspended solids, and a thing is unimportant except as it contributes to this end, other engineering principles being duly considered. The controversy regarding the use of synchronous motor-driven rectifiers by which the power for the treater is taken directly from the mains of the local power system, rather than the motor-generator rectifier giving each treater unit its own isolated electrical system, has led many to think that on the type of rectifier depends the success of the plant. It is in fact a relatively unimportant part of the plant and

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\* Assistant Research Engineer, American Smelting & Refining Co.

† Received Oct. 30, 1918.

represents a very small fraction of the investment; the recoveries in dust and fume are practically the same in each case. One large smelting concern has adopted the motor-generator type for all of its plants after years of experience with the synchronous-motor type. Its reasons are practically the same as those set down by Mr. Eschholz for his preference. Given more in detail they are:

1. An independent electric system prevents outside power-line conditions, like voltage fluctuations, from interfering with the operation of the Cottrell plant and eliminates the possibility of the Cottrell plant causing trouble for the power company. The latter, while not very probable, must be considered, for it is sometimes difficult to convince the transmission engineer that the make and break of the synchronous switch, which is the rectifier, does not introduce destructive voltage transients into his system like the well known phenomena attending high-tension switching. It is not surprising that the power company hesitates to take any chances for the small amount of load acquired.

2. An isolated electric system for each treater prevents the possibility of any disturbance in one treater being communicated to the other treaters through the transformers and rectifiers.

3. By means of the generator-field and the exciter-field regulations, the treater potential may be maintained very close to the critical disruptive value. This is considered essential for good work and can only be equaled when using a synchronous-motor type method by the use of an induction regulator with remote electric control, which is more complicated than a simple rheostat control in the generator field.

4. The motor generator is free from all the drawbacks to which the synchronous motor is subject, such as falling out of step and hunting.

Against these things is urged a slightly smaller first cost and approximately 86 per cent. power efficiency, as compared with about a 76 per cent. efficiency of the motor-generator set. In addition, if the synchronous-motor method is used, the large power system will tend to absorb the surges while the small generator circuit will tend to reflect and magnify any wave distortions that may be present. But if there are no wave irregularities, this function of a large power system is not necessary. Oscillograms taken on a large motor-generator Cottrell plant in Utah showed the treater and transformer circuits to be remarkably free from these surges. In this case, however, the generator wave was a true sine without any irregularities. Oscillograms taken on a large plant at Coram, Cal., where synchronous-motor rectifiers and induction regulators were used, showed that steep wave fronts and surges were present to a marked degree in spite of the absorbing qualities of a large power system. Oscillograms taken on three plants recently constructed, where motor generators were used, showed that under certain conditions of the load voltage transients in the high-tension side of the transformer circuit

became troublesome. In these plants the voltage wave was not a true sine, a number of small peaks or harmonics being apparent in the wave. The small peak that occurs just at the point of breaking contact is seen in the oscillogram to be manifest in the wave on the high side of the transformer greatly magnified, due to the steep wave front in the primary wave. From the foregoing it is a fair assumption that if the voltage wave of the generator is a true sine with no steep wave fronts there is no disadvantage due to surges in motor-generator rectifiers. This method, then, should not be condemned on account of a defect in a particular type of generator used. It seems better to correct the defect in the generator, although this defect has not proved as detrimental as might be supposed, for in every case known to the writer this surge, which makes itself manifest by arcing across a protective spark gap placed across the high-potential terminals of the transformer, decreases so as to be negligible as soon as the gas is in a state permitting good precipitation with either method. There are electrical remedies that often prove beneficial, such as absorbing this oscillating energy by inserting ballast resistance in the primary and secondary circuits or by shunting condensers across the primary circuit. Sometimes the inductive reactance of the circuit may be changed to advantage; and, too, the critical frequency of the system may be changed by changing the rectifier to contact to only one alternation per cycle without any loss in precipitation in the treater.

The disturbance manifest in the high-potential transformer circuit should not be confused with the treater surges that are manifest in sparking across between electrodes, although the latter often set up oscillations that cause surges in the former. The treater surges are said to have their origin in the trailing arc of the rectifier. At any rate, they are especially prevalent if the time of rectifier contact is carried into the neutral part of the wave. A very common cause is the deposition of a dry dust coating on the surface of the passive electrode. The sparks that jump across have all the characteristics of a condenser discharge and it is undoubtedly a case of disrupted dielectric due to being overcharged. It disappears as soon as the dielectric strength of the coating is destroyed.

A matter of considerable importance is that of conditioning the dust particle to assimilate a charge. This is usually done by introducing water into the gas by means of sprays. This does not necessarily increase the conductivity of the treater circuit in the usual sense for, at a number of plants, when water is introduced into a dry gas below the boiling point of water, the treater current is greatly reduced, with a corresponding increase in the treater potential and a much improved precipitation. This is especially noticed as the relative humidity of the gas is increased through the range of 30 to 50 per cent. It appears that a conductive coating has been imparted to the dust particle,



putting it in a condition to receive a charge, for previous to the admission of water the dust particles pass through the strong electric field of the treater unaffected, although the molecules of the gas are highly ionized. It would be interesting to know if the ionization that is known to accompany water spray enters in and if the agglomeration of the dust particles assists the precipitation.

The discussion of the paper has seemed to establish the fact that the mere presence of the water vapor is necessary without determining a reason. The theory of water being taken up by the particle was set aside on the assumption or statement that no more water was precipitated with the dust after the gas had been humidified than before. The writer does not concur in this assumption, for in his experience the dust precipitated becomes damper with increased relative humidity of the gas, until it becomes a mud above 70 or 80 per cent. relative humidity, in some cases. Some of this water may have been due to the fact that the passive electrode was cooler than the gas, making a supersaturated stratum adjacent to it, but in a number of cases the temperature of this electrode was such that no water could be condensed on it except by surface adsorption. In the very nature of things the dust particle must take up water from the aqueous vapor. By the well-known selective adsorption theory, aqueous vapor is known to be taken up by the surface of solids in preference to other gases, such as nitrogen and oxygen, and it is also well known that films of adsorbed moisture persist on the surface of solids at temperatures far above the boiling point. It is only necessary to cite the early work of Bunsen in his effort to remove the last traces of moisture from powdered glass or the exterior of glass tubes.

In regard to the theory of back ionization that has been advanced in the discussion, it is understood, by the writer, that if a dry dust coat covers the passive electrode there is a luminous glow reaching toward the active electrode, which results in reducing the gap in proportion to the length of the glow and this effect disappears as soon as the dielectric strength of the coating is destroyed by moisture. It seems that the glow is due to the fact that the dust particles forming the inner coating have not been able to give up their charge to the electrodes, and cause an electric field in opposition to the field due to the electrodes until they are made conductive and can give up their charge. This is a very promising theory and accounts for the fact that at a certain plant only 16 kv. can be maintained with an electrode spacing of 6 in. when the gas is dry, whereas a potential of 25 kv. or over can be maintained after the gas has been sufficiently humidified. It may also account for the automatic change in ratio between the transformer voltage and the voltage impressed on the treater when the change in the relative humidity of the gas occurs. It seems, however, that the phenomena of the glow can be accounted for

sufficiently by the theory of the overcharged gaseous dielectric of a condenser, advanced above.

A common fallacy is the assumption that most of the dust has a tendency to migrate to the passive electrode where the electric field is weakest. Experiments have convinced the writer that more dust is collected on the passive electrode because its greater surface affords a larger place for the particles to lodge and, speaking in terms of unit area, the tendency is really to go to the active electrode where the electric field is strongest, in spite of the electric wind in the opposite direction.

The writer does not share Mr. Eschholz's enthusiasm over the intermittent contact rectifier commonly used. It has a very poor regulation under varying conditions of the gas in the treater and many oscillograms show that the treater voltage often falls to one-half its maximum value between impulses. The rectifier does have the advantage of ruggedness and comparatively low first cost.

For the betterment of the process there is need of securing increased agglomeration of fume, a potential gradient best suited for ionization for an efficient charging of the dust particles, and a decrease in the cost of the treaters.

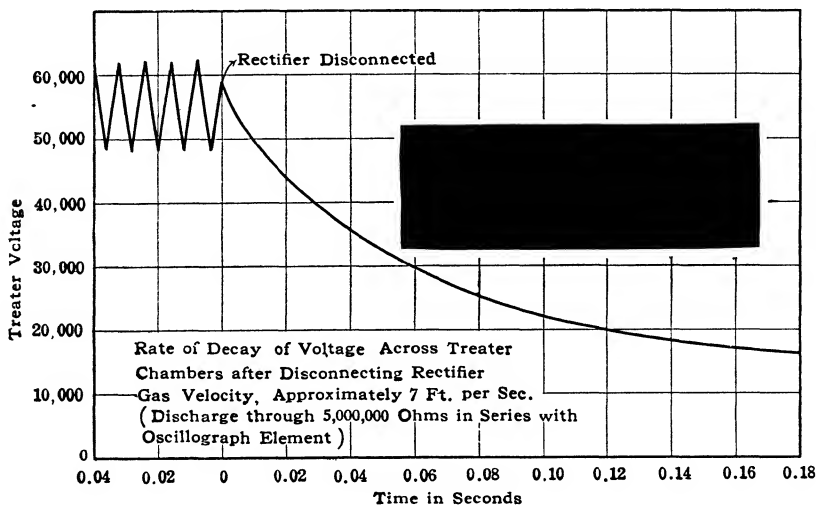
O. H. ESCHHOLZ (author's reply to discussion\*).—On reviewing the discussion it is apparent that the subject of power source has received the most consideration. When referring to the favored systems I clearly implied that the characteristics of separate generators for each treater (system A-1) had been commercially demonstrated for large as well as small installations. In view of the fact that a number of uncontrolled variables still exist in the applications of this process, it is with considerable relief that the precipitation engineer recognizes the advantageous operating characteristics of this system.

The operation of system B (power direct from industrial mains) has been demonstrated commercially on small treater installations only. This system has the manifest advantages cited in the paper and discussions. On large installations treating, let us say, 250,000 cu. ft. per min. or more, its commercial operation is still a matter of conjecture for the reasons given. The discussions of Messrs. Fischer, Rathbun, and Rosenblatt appear to support this conclusion. It is possible, as Mr. Fischer states, to obtain voltage control by changing the transformer primary taps. However, it has been my experience that, where frequent changes in voltage are necessary, operators distinctly prefer a rheostatic control because of the greater ease and rapidity of manipulation.

At the close of his very interesting discussion, Mr. Rathbun observed that "many oscillograms show that the treater voltage often falls to one-

half its maximum value between impulses." It is my opinion that where this is the case the voltage drop is due to excessive leakage, the most frequent cause of which is probably the "back ionization" discussed at length by E. R. Wolcott in the October, 1918, issue of the *Physical Review*.

The following figure illustrates the treater-voltage characteristics with the treater in good operating condition (slight leakage) before and after disconnecting the rectifier. Before opening the circuit the voltage wave is quite smooth, due to the fact that the treater chambers function as condensers and the leakage between the interval of rectifier arcs is slight. On disconnecting the rectifier, a typical condenser type of discharge is obtained through the resistance connected in series with the



oscillograph element, the time required for the voltage to drop to one-half of its maximum value being equal to approximately twenty times that of the interval between rectifier arcs.

In Mr. Rosenblatt's discussion of surges in the low-voltage circuit, a number of methods for absorbing the oscillating energy are mentioned. So far as I am aware, treater operators have completely overlooked the possibilities in the use of such apparatus. During a test made last summer, on my suggestion, a selective resistor, consisting of a coil of insulated cable wound on a solid iron core, when inserted in series with the primary circuit served to reduce circuit oscillations, increase the smoothness of the treater voltage, and reduce the rectifier noise owing to the fact that on the passage of high-frequency current the eddy-current loss in the iron increased, the apparatus functioning thereby as an equivalent resistor, the resistance of which increased with the current fre-

quency. It is anticipated that a still further improvement in operation may be secured by the use of a synchronous resistor. This consists simply of a resistance that may be inserted periodically in series with the primary circuit through the use of contacts and slip rings attached to the rectifier, the contact period being adjusted so that the resistance is placed in series with the primary circuit only during the interval between the breaking of the arc tail and the subsequent formation of the arc. It is my opinion that the conflicting experience encountered in the use of either supply system is almost entirely due to the surges in the low-tension circuit, and their satisfactory elimination will only be secured with the adoption of some rugged and economical means for absorbing the oscillating energy.

## Condensation of Zinc from its Vapor

BY CHARLES H. FULTON,\* CLEVELAND, OHIO

(Colorado Meeting, September, 1918)

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### INTRODUCTION

The study of the condensation of zinc from its vapor was undertaken to shed light on certain problems encountered in large-scale electric zinc-furnace work recently conducted. It is a matter of common knowledge that one of the disadvantages of the arc type, electric zinc furnace, is the production of a large amount of blue powder and proportionately little liquid spelter, for, as stated by Stansfield,<sup>1</sup> "A notable defect in the electric smelting of zinc ores is the difficulty experienced in obtaining the distilled zinc in the liquid state; . . . . . when zinc ores are smelted electrically, very little liquid metal is commonly obtained, nearly all of the zinc being in the state of powder."

This difficulty was not encountered in the electric furnace used,<sup>2</sup> the main difficulty being the destruction of the fire-brick lining of the large condenser, in certain parts, and the formation of some oxide and dross coatings on the condensing surfaces. The reason for these difficulties could readily be explained by the infiltration of air whenever the condenser was changed from one retort base to the other, the zinc absorbed in the fire-brick forming oxide and causing rupture by its expanding volume. Two facts, however, tended to disprove this theory for the rupture of the fire-brick lining: first, the large distilling retort, lined with fire-brick, which was shifted from charge to charge on alternate bases at temperatures between 1200° and 1300° C., fully exposed to the air, showed no signs of disintegration whatever; second, marked disintegration in the condenser occurred only in places that were at tempera-

\* Professor of Metallurgy, Case School of Applied Science.

<sup>1</sup> A. Stansfield: "The Electric Furnace," Ed. 2, 325. N. Y., McGraw-Hill Book Co., 1914.

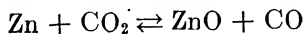
<sup>2</sup> U. S. Patent Office Official Gazette (1917) 234, 1068; 242, 316, 318. 1,213,180; 1,242,337; and 1,242,341.

tures ranging between 550° and 450° C. The fire-brick used in the condensers was a Mexico, Mo., brick of excellent grade, but containing numerous "iron spots." An examination of ruined brick and tile showed that these iron spots had been filled with fine sooty carbon, and that rupture was due to the increase of volume due to this deposition. This is a comparatively rare, but well-known phenomenon in the iron blast-furnace, and is clearly described by Frank Firmstone.<sup>3</sup>

The action is due to the breaking up of carbon monoxide into carbon dioxide and carbon at the temperature at which this reaction is most active (600–450° C.), in the presence of the catalytic agent iron (reduced from the iron oxide by the CO).

This discovery introduced a new problem into the condensation of zinc vapor in large condensers: Is enough carbon dioxide formed by the breaking down of carbon monoxide to interfere seriously with condensation? While practically no blue powder had been formed in the experiments, it was thought that the dross and rock oxide might be due to the carbon dioxide; although the infiltration of air was sufficient to explain their presence, nevertheless the CO<sub>2</sub> might be responsible for it in the absence of oxygen. In the circumstances, analyses of gas taken at the exit of the condenser were considered of no particular value, since, if the CO<sub>2</sub> acted on the metallic zinc vapor to reoxidize it, more CO would be formed, and the fact that there had been a decomposition of CO could not be detected. That the decomposition of CO has an effect on the condensation of zinc has been suggested by Stansfield.<sup>4</sup>

A search of the literature bearing on condensation revealed practically no facts, nearly all of it being speculative. Data on the composition of gases arising from the distillation of zinc ore are practically limited to those given by Ingalls,<sup>5</sup> which are also stated by R. G. Max Liebig.<sup>6</sup> No record could be found for the equilibrium conditions of the reaction



except general statements, which are referred to later (p. 293).

## DISTILLATION PRODUCTS FROM REDUCTION OF ZINC ORE

### *Method of Investigation*

Experiments were conducted under rigorous conditions to determine the composition of the gas and the condition of the condensed zinc

<sup>3</sup> F. Firmstone: An Example of the Alteration of Fire-brick by Furnace Gases. *Trans.* (1903) **34**, 427.

<sup>4</sup> *Op. cit.*, 330.

<sup>5</sup> W. R. Ingalls: "The Metallurgy of Zinc and Cadmium," 204. N. Y., *Engineering and Mining Journal*, 1903.

<sup>6</sup> "Zink und Cadmium und ihre Gewinnung aus Erzen und Nebenprodukten," 380. Leipzig, Spamer, 1913.

from the distillation of zinc ore. For this purpose porcelain and quartz tubes were used, having been demonstrated to be tight and refractory enough for the temperatures employed. Zinc at high temperatures has great affinity for oxygen, removing traces of oxygen from gases, just as copper does. For this reason it was necessary to insure that oxygen

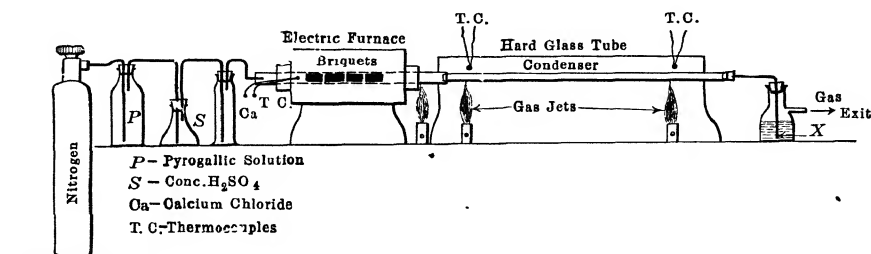


FIG. 1.—APPARATUS FOR INVESTIGATING PRODUCTS FROM DISTILLATION OF ZINC ORE.

was excluded from the system if the relation between the condition of the zinc and the normal gas composition was to be judged. The arrangement of the experimental apparatus is shown in Figs. 1 and 2. The retort part of the tube was heated in a horizontal, granular-resistance, tube furnace similar in design to the vertical tube furnace used by the author in previous work.<sup>7</sup> The condenser part of the tube was heated,

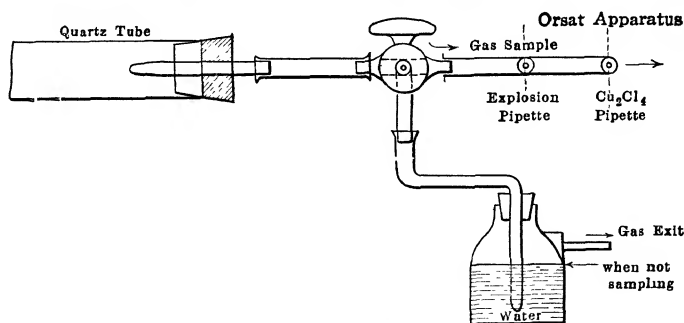


FIG. 2.—DETAIL OF GAS DISCHARGE FROM CONDENSER WHEN USING ORSAT APPARATUS.

when desired, by a gas-fired combustion furnace. The charge for distillation was in the form of briquets,<sup>8</sup> made of 100 parts Missouri calamine ore, 80 parts of crushed coke, and 20 parts of hard coal-tar pitch. These briquets were approximately 1 in. (25.4 mm.) in diameter and 1¾ in. (44.4 mm.) long and were quartered longitudinally to fit inside the tube. They had previously been baked at a temperature of about 500°C., to drive out the most readily volatile constituents, and contained 18.5

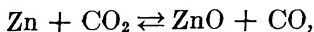
<sup>7</sup> C. H. Fulton: Constitution and Melting Points of a Series of Copper Slags. *Trans.* (1912), **44**, 769.

<sup>8</sup> U. S. Patent Office Official Gazette (1916) **229**, 342. No. 1,193,680.

per cent. zinc. The ore contained 39.5 per cent. zinc, 35 per cent. insoluble matter, considerable water, and some carbon dioxide. In composition the briquets were the same as those used in the large electric furnace at East St. Louis, Ill., and do not differ from the charge of the common zinc retort. The gases from the distillation of this material should resemble zinc-retort gas after the charge has been thoroughly dried and heated to about 500° C.

Oxygen was excluded by the following procedure: the system was thoroughly tested to insure tightness at the temperature used, the quartered briquets were then introduced into the tube, the system closed, and commercial nitrogen (purified by passage through water, pyrogallie solution, conc. sulfuric acid, and calcium chloride) was passed till all air was considered displaced (usually about 5000 c.c.). The furnace was then heated, and, when the gas flow from the distillation was free and it was thought that the nitrogen was displaced, gas samples were taken and analyzed. The sample was taken by inserting the exit tube X, Fig. 1, which was drawn to a small opening, directly into the water-filled rubber connection of the gas burette. Gas analysis was made with the gas burette and Hempel pipettes, great care being taken in regard to temperature conditions, drainage, and connections. It was not considered necessary to employ the exact methods of analysis, using mercury. In some of the experiments an Orsat apparatus was used, directly connected to the exit tube by means of a two-way cock, as shown in Fig. 2.

Approximately 30 gm. of briquet material were used in each experiment, yielding about 3000 c.c. of gas. When the gas flow ceased, the furnace was cooled, nitrogen being passed meanwhile. In later work it was found that when nitrogen, or other gas, was passed through pyrogallie solution, or through both pyrogallie and phosphorus, traces of oxygen remained, which acted on zinc at a temperature between 500° and 700° C. In the above experiments, any traces of oxygen would perhaps be taken up by the carbon of the briquets, but the very slight coatings found on some zinc globules may have been due to this small amount of oxygen; in experiments, described later, conducted in apparatus made entirely of graphite, but not tight against air, some of the zinc was decidedly coated. It is evident from the equilibrium conditions of the reaction



in the presence of carbon, that, when the temperature will permit the formation of dioxide from carbon and oxygen, zinc oxide may form simultaneously.

Table 1 gives the composition of the gases from the distillation of the zinc-ore briquets, under the conditions above described. In all of the experiments using nitrogen as the displacing gas (No. 7 to 16



TABLE 1.—*Products from Distillation of Zinc Ore*

Number	Stage of Distillation	Purification Train for Displacing Gas	Gas Composition							Remarks and Notes
			CO <sub>2</sub>	CO	O <sub>2</sub>	CH <sub>4</sub>	H <sub>2</sub>	N <sub>2</sub>		
7a	Beginning	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub>	0.65	83.7	Tr.	14.9			39-in. porcelain tube of which 16 in. was heated as a condenser. Zinc mirror bright.	
8a	Beginning	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyrogallic	1.10	78.2	2.2	18.6				
8b	End	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyrogallic	2.5	79.2	1.3	2.2	6.2	8.4	39-in. porcelain tube of which 16 in. was heated as a condenser. Zinc mirror bright.	
11a	Very beginning, 950°C.	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyrogallic	13.8	40.4	0 0	4 0	27 3	14 5		
11b	Middle, 1150°	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyrogallic	0.0	78.3	0.0	2 1	13.7	6 1	5½-in. porcelain tube, 21 in. long + 29 in. of ½-in. glass tube not heated. Zinc mostly bright	
11c	End. Temp very high, 1400°	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyrogallic	2.2	92.9	0 0	0.7	1.8	2.4		
12a	Beginning, 1000°	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyro- gallic renewed	0.8	70 8	3.5?	1 0	7 0	16 5	39-in. porcelain tube, no part heated as condenser. Zinc bright; few globules coated.	
12b	1300°	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyro- gallic renewed	0.95	81.4	0 0	1 3	7 6	8 9		
12c	End, 1500°	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Pyro- gallic renewed	6.6	59 0	0 0	0 0	0.0	34 5	Gas flow very slow at end	
16a	Beginning, 1050°	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Fresh pyro- gallic	0 65	61.6	0 0	10 3	10 0	17.3	¾-in. quartz tube 24 in. long. Zinc bright, some with refraction color film.	
16b	Middle	Conc. H <sub>2</sub> SO <sub>4</sub> CaCl <sub>2</sub> Fresh pyro- gallic	0.65	79.5	0 0	8.5	6 1	5 3		
18a	Beginning	Conc. H <sub>2</sub> SO <sub>4</sub> , H <sub>2</sub> O, pyro- gallic, CaCl <sub>2</sub>	1.30	77.6	0.0	4.3	15.1	4 4	¾-in. quartz tube 24 in. long. No condenser. Coated zinc.	
18b	End. Temperature high	Conc. H <sub>2</sub> SO <sub>4</sub> , H <sub>2</sub> O, pyro- gallic, CaCl <sub>2</sub>	0 30	71.7	0.0	4 9	14.3	8.8		
19	Middle	Conc. H <sub>2</sub> SO <sub>4</sub> , H <sub>2</sub> O, pyro- gallic, CaCl <sub>2</sub>	0 0	70.3	0.0	3 3	20.7	5.4	¾-in. quartz tube 24 in. long. No condenser. Coated zinc.	

NOTES.—The displacing gas was commercial nitrogen in tests No. 7 to 16, incl.; and hydrogen in tests No. 18 and 19. For analysis of the gas, the burette and Hempel pipettes were used in tests No. 7 to 12, incl.; and the direct-connected Orsat apparatus in tests No. 16 to 19, incl.

incl.) the zinc was essentially mirror bright, a slightly coated globule sometimes being found. In the experiments in which hydrogen was the displacing gas (No. 18 and 19), the zinc was coated with the characteristic gray coat, and in some cases zinc oxide crystals were found.

### *Nitrogen*

In the experiments with nitrogen as the displacing gas (No. 7 to 16, incl.), it will be noted that the distillation products contain appreciable percentages of nitrogen. The quantity is too great to be attributed to nitrogen left in the tube, since practically all of this should be displaced by the gas stream from the distillation. To make certain of this point, hydrogen (made from zinc and dilute sulfuric acid and purified as indicated in Table 1) was used as the displacing gas. The distilled gases still contained nitrogen in quantity; hence it must be concluded that nitrogen is a normal gas product of zinc distillation. This is also true of hydrogen, although R. G. Max Liebig,<sup>9</sup> ascribes both of these gases to diffusion into the retort from the combustion chamber. That diffusion into the retort occurs is unquestionable, as will be shown later; but in the present investigation it seems certain that this possibility was excluded.

In the distillation of coal and the manufacture of coke in byproduct ovens, nitrogen is recognized as a normal constituent of the resultant gas. O. Simmersbach<sup>10</sup> has shown that the gas coming from Kopper's ovens at the end of the coking period, at a temperature of about 1100° C., contains 15 to 17 per cent. nitrogen, and 60 to 70 per cent. hydrogen, the nitrogen being higher at the end of the coking period than at any other time. Coke always contains nitrogen, as high as 1.84 per cent. being found in English coke.<sup>11</sup> This nitrogen is not expelled completely except at very high temperatures. In experiment 12c, at the very end of the distillation, at a temperature of about 1500° C., the nitrogen was found to be high. This sample was carefully taken when the gas flow had become very slow, and represents the product at the very end of distillation, or even after distillation was complete. The high nitrogen is not to be explained by the supposition that more nitrogen is given off at this time, but rather by the fact that practically no other gases are being evolved, nitrogen being the last gas to be given off. In discussing the presence of nitrogen in the distillation gases, it must be borne in mind that hard coal-tar pitch was used as the binder for the briquets. Since the briquets

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<sup>9</sup> *Op. cit.* (1913).

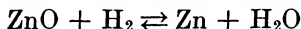
<sup>10</sup> O. Simmersbach: Untersuchungen über die Temperaturverhältnisse im Koksofen. *Stahl und Eisen* (June 14, 1914) **34**, 954; quoted by F. H. Wagner: "Coal and Coke," 334. N. Y., McGraw-Hill Book Co., 1916.

<sup>11</sup> H. O. Hofman, "General Metallurgy," 235, quoting authorities. N. Y., McGraw-Hill Book Co., 1913.

were baked at a temperature of about 500° C. before distillation, this pitch was converted into a coke, but still contained some heavy hydrocarbons, which break down on further heating. Pitch contains somewhat less nitrogen than coke. It is evident that the remarks on nitrogen also apply to the distillation process when coal is employed as the reducing agent.

### *Hydrogen and Hydrocarbons*

The reduction of zinc oxide by hydrogen according to the reaction



undoubtedly takes place at a markedly lower temperature than the reduction by carbon, and in this experiment reduction by the hydrogen began before the gas flow due to the carbon reaction became pronounced. In the hydrogen reduction, equilibrium conditions demand increasing concentration of hydrogen with increasing temperature; hence as the hydrogen supply decreased, since it was cut off when the furnace was heated, the reaction reversed itself, with the formation of zinc oxide. At the end of the experiment, water was found in the cooler portions of the tube. The action of hydrogen on zinc oxide was investigated by Percy<sup>12</sup> and Deville,<sup>13</sup> both of whom found that to reduce zinc oxide by hydrogen it is necessary to have a rapidly moving stream of hydrogen; that is, the water produced by the reaction must be removed and not allowed to attain any appreciable concentration, if reoxidation is to be avoided.

The presence of methane and hydrogen in distillation gases is well known. When coal is used as the reducing agent, they represent the decomposition products of hydrocarbons. This is also true when coke and pitch are used, although the amount of hydrocarbons in the reduction material is much smaller. The two gases are most abundant at the beginning of the distillation, and disappear toward the end. In the experiments described, the briquet material occupied a considerable length of the heated tube, and, as all of it was not at the same temperature simultaneously, methane and hydrogen were evolved from the cooler ends of the tube even toward the close of the distillation period. This feature may be likened to the condition in the common retort, in which the charge is gradually heated from the outside.

In view of the action of hydrogen in the reduction of zinc oxide, the question arises as to the effect, on the zinc, of the hydrogen normally present in distillation gases. This hydrogen is evolved from the charge in the presence of a large excess of carbon, and if any water does form from the reduction of zinc oxide by hydrogen, it is immediately reduced again to

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<sup>12</sup> John Percy: "Metallurgy," 535. London, John Murray, 1861.

<sup>13</sup> H. Sainte-Claire Deville: Note sur la Réduction de l'Oxyde de Zinc et des Alcalis. *Annales de Chimie et de Physique*, Ser. 3 (1855) 43, 479.

hydrogen, by the carbon, with the formation of  $\text{CO}$ ,  $\text{CO}_2$ , or both, depending on the temperature. According to a summary by Roscoe and Schorlemmer,<sup>14</sup> at temperatures of about  $1000^\circ \text{C.}$ , the reaction between carbon and water vapor produces carbon monoxide and hydrogen, with very little carbon dioxide. As the temperature is lowered, the production of carbon dioxide increases, until at  $600^\circ \text{C.}$  only carbon dioxide and hydrogen are produced.

The reduction of zinc from ores, by carbon, begins at temperatures ranging from  $900^\circ$  to  $980^\circ \text{C.}$ , depending on the kind of ore. These figures were obtained by the author some time ago and represent the temperatures at which zinc first becomes visible in the ignited distillation gases from small vertical graphite retorts, heated uniformly to the gas exit in an electric tube furnace. The exact figures were: Mascot, Tenn., ore,  $910^\circ$ ; Cañon City, Colo., concentrate,  $940^\circ$ ; roasted Joplin blende ore,  $940^\circ$ ; Burma, zinc-lead ore,  $980^\circ$ .<sup>15</sup> The reduction of zinc by hydrogen, as previously stated, occurs at a notably lower temperature, although no definite figures are available.

#### *Carbon Monoxide and Dioxide*

At the reduction temperature of zinc oxide by carbon, carbon monoxide is the normal reaction product, since, in the presence of an excess of carbon, at this temperature, only very small quantities of carbon dioxide can exist. If, however, zinc oxide is reduced by hydrogen or by methane, evolved from the charge at temperatures below  $800^\circ \text{C.}$ , the reaction products will consist of water, carbon monoxide, and *carbon dioxide* in appreciable quantity. It is well known that carbon dioxide in certain concentration is highly undesirable in zinc metallurgy, and some definite figures on this subject are given later in this paper. In the author's opinion, the high proportion of carbon dioxide in the distillation gases at the beginning is due to the above described reactions, although some of it comes from the reduction of iron oxides in the ore. Any zinc reduced by hydrogen or methane at temperatures below the carbon reaction will be in contact with distillation gas relatively high in carbon dioxide, and will suffer oxidation, with the consequent production of blue powder. From this standpoint, reduction coal which liberates appreciable quantities of hydrocarbons at relatively low temperatures is undesirable. Hydrocarbons and hydrogen evolved after the carbon reduction temperature has been reached are not harmful.

<sup>14</sup> Sir H. E. Roscoe and C. Schorlemmer: "Treatise on Chemistry," Ed. of 1905, 1, 793. Macmillan, London and New York.

<sup>15</sup> See also figures by F. O. Doeltz and C. A. Graumann, Versuche über die Reduktion von Zinkoxyd. *Metallurgie* (1907) 4, 290; and those quoted by W. R. Ingalls, "Metallurgy of Zinc and Cadmium," 2d ed. (1906), 198, and W. McA. Johnson, *Engineering and Mining Journal* (1904) 77, 1045.

Carbon dioxide in excess of 1 per cent. is not found in the gases distilled above the carbon reduction temperature;\* below that temperature as high as 13.8 per cent. was found (experiment 11c, and others not recorded here). At the end of the distillation, the proportion increases, particularly when the temperature is high (above 1400°), the monoxide suffering decomposition into carbon dioxide and carbon.<sup>16</sup> This reaction may have an effect on the zinc when smelting in an arc electric furnace, in which very high local temperatures prevail. Carbon dioxide under 1 per cent. has no effect on zinc vapor either above or within the condensation temperature range, as is shown later in this paper.

### *Oxygen*

Oxygen, except in very small quantities, is not a normal constituent of the distillation gases. A series of experiments was made to obtain information as to the accuracy of the gas apparatus used for estimating small quantities of oxygen in such gases as commercial nitrogen. Oxygen to the proportion of 0.5 per cent. and over was readily determined, but when testing nitrogen, after purification with pyrogallic solution or phosphorus, it was difficult to obtain results which could not be accounted for by slight changes in temperature, absorption by the collecting water, or absorption of other gases by the solutions employed, although heated copper showed that small amounts of oxygen were still present. It is possible that oxygen in very small quantities was present in the distillation gases, but this was improbable since zinc vapor has a very great affinity for oxygen. The oxygen recorded in samples 8a, 8b, and 12a was probably due to defective sampling.

### *Condition of Zinc*

The zinc condensation was sharply localized in the tube almost immediately beyond the distilling briquets. In experiments 7 and 8, about one-half of the tube was heated uniformly by gas burners to 620° to act as a condenser, but only very small amounts of zinc were found in it, practically all of it being condensed as a mass of globules in the roof of the tube and as a little lake in the bottom beneath these, all within the space of about 1.5 in. (38 mm.). This form of condensation is to be expected from the vapor-tension curve of zinc, which shows that, in a mixture of equal parts of zinc vapor and carbon monoxide, 87 per cent. of the zinc will be condensed within the temperature range of from 865° to 750° C.

In these experiments the heating conditions were such that, immediately beyond the distillation space, the temperature rapidly dropped to between 700° and 800°, so that the zinc, for the most part, condensed

<sup>16</sup> V. Meyer and C. Langer: *Pyrochemische Untersuchungen. Berichte der Deutschen Chemische Gesellschaft* (1885) **3**, 134.

\* See author's reply to discussion, p. 301. Ed.

here. The tubes were  $\frac{7}{16}$  in. (11 mm.) and  $\frac{3}{4}$  in. (19 mm.) diameter; and the gas flow being relatively slow, about 30 c.c. per minute, the diffusion of heat into the walls permitted a sharply localized condensation of the zinc. The zinc globules were removed by a wire rod, but the little lakes of zinc adhered so firmly to the tubes that it was necessary to break them to examine the metal. In experiments 7 to 16 inclusive, the zinc was practically all mirror bright; occasionally a globule was found with a slight brown or grayish coat, not discernible to the eye, but plain under the microscope. Adhering to this coated globule were sometimes many small mirror-bright globules. At a certain period of the distillation the gas composition may have been such as to attack certain globules, or the coating may have been due to some substance, as cadmium sulfide or carbon. The coating was not the typical blue-gray coat formed when  $\text{CO}_2$  or  $\text{O}_2$  is present. Some of the globules had assumed definite crystal structure in solidifying. In that portion of the tube on the side where the displacing gas entered was sometimes found a faint yellow-white film which, when dissolved in dilute  $\text{HCl}$ , gave the odor of hydrogen sulfide, and, after oxidation with nitric acid, gave a distinct reaction for sulfate. This coating consists probably of a mixture of zinc oxide and cadmium sulfide; the zinc oxide was probably formed by contact of zinc vapor with a small proportion of oxygen remaining in the displacing gas, while the cadmium sulfide probably vaporized as such from the charge. According to Olsen,<sup>17</sup> cadmium sulfide sublimes at  $980^\circ \text{C}$ . In experiment 11, a small amount of zinc frost was found; it consisted of bright feathery interlaced crystals of zinc, and evidently had passed directly to the solid state from the vapor. It was not coated. In some of the experiments, on opening the tube the odor of hydrocyanic acid was distinctly noticeable.

#### *Conclusions from Preceding Experiments*

When a charge of zinc ore and reducing agent, consisting of coke and the coked residue of hard coal-tar pitch, is submitted to the distillation process in a tight system free from oxygen, the zinc is distilled from the charge and then condensed into bright metal with the presence, only occasionally, of a foreign material which might interfere with coalescence; this is probably a sulfide, and carbon, but it occurs in such small amounts as to be technically negligible. Under proper temperature conditions the condensation is sharply localized, and no so-called blue powder is formed.

The gases consist of carbon monoxide, carbon dioxide, methane, hydrogen, and nitrogen. The carbon dioxide is highest at the beginning, but rapidly decreases to below 1 per cent.; toward the end of the distillation, methane and hydrogen disappear, and carbon monoxide increases un-

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<sup>17</sup> Van Nostrand's *Chemical Annual* (1918), 147.

til it composes by far the greater part of the gas. Nitrogen is persistently present in considerable quantity. Carbon monoxide suffers no appreciable decomposition into carbon dioxide and carbon in passing through the condenser when heated to between 500° and 700° C. This point is again referred to in this paper.

### PRELIMINARY INVESTIGATIONS

The experiments recorded above, and the development of the apparatus in which they were carried out, were preceded by considerable preliminary work which revealed the fact that experimental investigation of zinc at high temperature presents difficulties, in that zinc has a very great affinity for oxygen and other oxidizing gases, even in minute quantities, and that if reliable results are to be obtained these must be excluded. An air-tight apparatus is therefore essential. Before this was achieved, some interesting results were obtained with apparatus that was supposed to be tight, but was found not to be so at the temperatures used.

#### *Reduction in Carbon Tube*

Since excess of carbon is normal in the usual distillation process, it was natural to try a hard carbon tube as the retort in an electric furnace. A hard, dense carbon tube,  $\frac{7}{8}$  in. (22 mm.) diameter with  $\frac{1}{8}$ -in. (3-mm.) walls, tight against pressure by blowing, and showing no escape of gas bubbles under water, was used. Other conditions of the experiment, the displacing gas, its purification, and the sampling, were the same as those described in Table 1. A hard glass tube,  $\frac{1}{2}$  in. (12.7 mm.) diameter and 36 in. (91.4 cm.) long, heated to 700° to 500° C. along its length, was used as condenser. The zinc obtained from these experiments was found partly in the carbon and partly in the condenser tube, being rather widely disseminated. It was heavily gray-coated, even in the carbon tube, although it had run together quite well. Zinc dust and some oxide were found in the condenser, and zinc dust was carried into the water bottle. The gas had the following composition, in percentage by volume:

Number	CO <sub>2</sub>	CO	O <sub>2</sub>	CH <sub>4</sub>	H <sub>2</sub>	N <sub>2</sub>
1	0.81	58.3	0	5.0	1.3	34.6
2	0.9	51.1	..	48.0		
3	3.0	58.4	..	5.5	2.4	30.6

The system was practically tight, and during the passage of the displacing gas gave no evidence of leaks. At the end of the experiment, the

tube was found to be slightly porous when subjected to considerable pressure. The results are therefore explained by the diffusion of air into the tube, as is evident from the high nitrogen contents. During the experiment the interior of the tube was under a pressure of about 4 in. of water. It is interesting to note that zinc was oxidized simultaneously with carbon, by the infiltrated oxygen.

### *Reduction in Graphite Retort*

In order to work with larger quantities of briquet material, a graphite retort was constructed of Acheson graphite, to the design and dimensions shown in Fig. 3. The apparatus included the retort *R*; the condenser

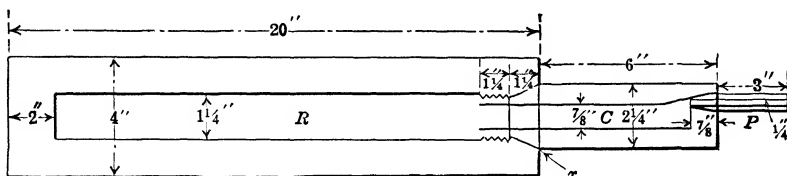


FIG. 3.—GRAPHITE RETORT AND CONDENSER.

*C*; and the prolong *P*. The condenser was fitted to the retort by a screw-cone joint made tight with stove cement, and the prolong was attached to the condenser by a tight cone joint. The condenser and retort were heavily coated with Johns-Manville high-temperature cement. A glass tube was cemented into the prolong, and the distillation gases were passed through water. The apparatus was heated to the distillation temperature in a large muffle. The gas flow was fast and free during most of the distillation, escaping against a head of 5 in. (12.7 cm.) of water. The sample was taken by inserting the discharge tube directly into the rubber connection of the gas burette.

The charge consisted of 334 gm. of briquets, which, after distillation, weighed 221 gm., a loss of 33.8 per cent. indicating complete distillation, from long experience with this type of material. The amount of zinc recovered was 23 gm., the loss being 38.8 gm.; this was not lost with the gases, for practically none was evident here, but must have been due to absorption and diffusion. The gas composition was as follows, in percentage by volume:

Number	Temp. °C.	CO <sub>2</sub>	O <sub>2</sub>	CO	CH <sub>4</sub>	H <sub>2</sub>	N <sub>2</sub>
1	1200	0.7	0.0	35.6	0.0	0.0	63.7
2	1300	1.4	0.0	72.5		26.1	
3	1350	0.0	0.0	52.6	0.0	0.0	47.5



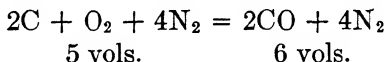
The temperatures were taken by a Pt-Rh thermocouple on the outside wall of the retort at the middle, the retort being uniformly heated throughout its length. The absence of hydrogen and methane is explained by the fact that the gas samples represent the latter part of the distillation and these gases had been expelled.

### *Experiment with Blue-powder Briquets*

Another experiment, in the same apparatus, using briquets made from coke, coal-tar pitch, and high-grade blue powder, gave interesting results. Briquets of this composition should yield very little gas, since there is practically no reduction, and zinc vapor is the only product of the distillation. There was a constant flow of gas from the retort during the distillation, and at a temperature of about 1000° C. its composition was as follows, in percentage by volume:

Num- ber	CO <sub>2</sub>	O <sub>2</sub>	CO	CH <sub>4</sub>	H <sub>2</sub>	N <sub>2</sub>
1	8.6	0.0	28.4	0.0	0.0	62.9
2	3.1	0.0	31.1	0.0	0.0	65.8

This is essentially the composition of producer gas, and is explained by the infiltration of air into the retort, the oxygen uniting with the carbon of the retort. The fact that the gas flows freely from the retort is explained by the increase in volume due to the reaction, as follows:



The zinc obtained in these experiments was partly bright, but mostly gray coated. Considerable blue powder was found in the condenser, although this was heated to the proper temperature by conduction from the retort. The oxidation of the zinc can be explained only by the action of oxygen, since in most cases the carbon dioxide was not high enough to react with zinc, as will be shown later.

### *Conclusions from Preliminary Experiments*

The results obtained with the carbon tube and the graphite apparatus point to some interesting facts in distillation. At first sight, it would appear that such an apparatus must be tight, particularly the retort, which is made of dense graphite, with walls 1.375 in. thick, but experience proved that diffusion took place freely. Further, in spite of the fact that the retort material was carbon, the zinc was partially oxidized and blue powder was formed, although the temperature conditions for

proper condensation to liquid zinc were maintained. This shows that when oxygen comes in contact with zinc vapor, even in the presence of carbon, at a temperature above 1000°, oxidation occurs. From the standpoint of good condensation of zinc, a tight distillation system is essential; not only must there be no openings into the system, but the material from which it is constructed must be proof against the powerful diffusion forces at work. It seems probable that much of the difficulty experienced in recovering a high percentage of liquid spelter, in the common retort process, is due to porous retorts and condensers. That there is considerable gas pressure outward from the retorts and condensers means but little when diffusion forces are at work.

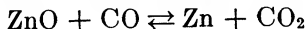
These remarks apply with still greater force to the electric zinc furnace of the arc type, and it would be remarkable if such furnaces as have been used did not yield large percentages of blue powder. This subject is again referred to later in this paper.

#### EQUILIBRIUM OF REACTION $\text{Zn} + \text{CO}_2 \rightleftharpoons \text{ZnO} + \text{CO}$ IN TEMPERATURE RANGE OF CONDENSATION

That zinc is oxidized by carbon dioxide is well known, but the author was unable to find definite data in the literature. The work described in the first part of this paper proves that the "normal" gases arising from distillation, and present during the condensation, containing up to approximately 1 per cent. or slightly more of  $\text{CO}_2$ , have no deleterious effect on condensation. Under certain conditions, more  $\text{CO}_2$  may be present, as in the case of early reduction by hydrogen or hydrocarbons, or by the decomposition of carbon monoxide.

Zinc oxide is reduced to zinc by carbon monoxide at 600° C.<sup>18</sup> and zinc is oxidized by  $\text{CO}_2$  at red heat, with the formation of CO, this gas constituting over 90 per cent. of the gas issuing from the experimental tube. One of the methods of making CO gas, given in laboratory manuals, is to pass  $\text{CO}_2$  over heated zinc.

A. Lencauchez<sup>19</sup> states that at white heat ZnO is reduced to Zn by CO, but that as the vapor and gases of the reaction pass to the cooler portions of the tube, the  $\text{CO}_2$  reoxidizes the zinc. He also states that blast-furnace gas containing 24 per cent. CO and 12 per cent.  $\text{CO}_2$  readily reduces ZnO at white heat, but as the temperature falls to cherry red (which he states is equivalent to 1200° C.) the  $\text{CO}_2$  reoxidizes the zinc vapor. These statements indicate that in the reaction



<sup>18</sup> Doeltz and Graumann: *Ibid.*

<sup>19</sup> A. Lencauchez: *Métallurgie du Zinc de la Condensation des Vapeurs de Zinc dans les Appareils Soufflés. Mémoires de la Société des Ingénieurs Civils* (1877), 568, 580.

equilibrium requires an increasing concentration of  $\text{CO}_2$  with increasing temperature, and a decreasing concentration of  $\text{CO}$ , the reducing agent, with increasing temperature. This is the opposite condition to that prevailing in the  $\text{ZnO}$ -hydrogen reaction, and most other reactions involving the reduction of metallic oxides by  $\text{CO}$ . In the experiments described below there was nothing to indicate that the nature of the  $\text{ZnO}$ - $\text{CO}$  reaction differed in this respect from the  $\text{ZnO}$ -hydrogen reaction.

### *Method of Investigation*

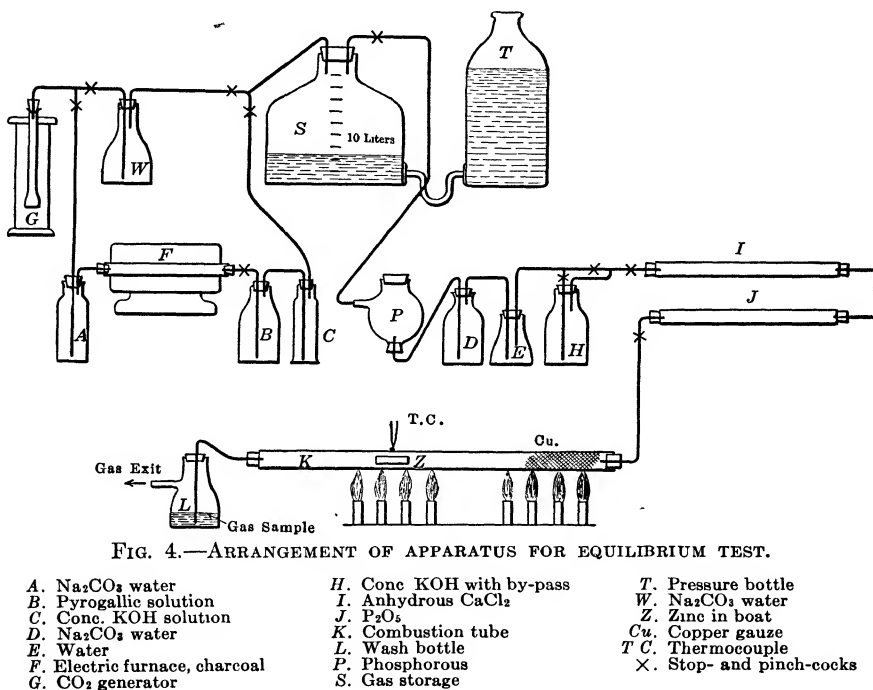
A piece of pure<sup>20</sup> bright zinc, in a porcelain boat, was placed in a  $\frac{1}{2}$ -in. (12.7-mm.) hard glass tube 36 in. (91.4 cm.) long, heated in a gas combustion furnace having a mica sheet immediately over that part of the tube containing the zinc, so that the condition of the metal could be readily observed during the experiment. Purified mixtures of  $\text{CO}$  and  $\text{CO}_2$  were passed, and the effect on the zinc was noted. The temperature was measured by a Pt-PtRh thermocouple, having its junction placed directly over the center of the zinc and touching the outside of the glass tube. The temperature could be held steadily at any point in a range from  $500^\circ$  to  $700^\circ$  C. The vapor tension of zinc is such that, within the temperature range adopted, zinc vapor in considerable quantity formed in the tube and was in contact with the stream of gas. This zinc vapor condensed in the cooler portions of the tube, and in part on the cooler sides of the porcelain boat. The deposited metal could afterward be examined under the microscope, which made it easy to distinguish sharply between bright metal, oxide, and coated metal. When oxide formed, it would partially deposit on the upper side of the tube immediately above the head of the porcelain boat, and also further along; but when the composition of the gas was such that no oxide formed, this head deposit would be absent, and bright zinc metal would condense in globules, and as a mirror, above the end of the boat and further back in the cooler portion of the tube.

Fig. 4 shows the arrangement of the apparatus as used in the final successful experiments, after considerable work had been done to ascertain the proper conditions for success. At first, carbon monoxide, made from potassium ferrocyanide and sulfuric acid, was passed directly into the gas storage *S*, which also received the carbon dioxide made from pure marble and dilute  $\text{HCl}$ . The gas storage bottle was graduated and any desired mixture could be made. The mixed gases were purified by passage through water, phosphorus, conc. sulfuric acid, and anhydrous calcium chloride. Under these conditions, carbon monoxide free from

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<sup>20</sup> The analysis was: Pb, 0.04; Fe, 0.025; Cd, 0; As, tr.

carbon dioxide formed coatings on the zinc, and the difficulty was ascribed to small quantities of  $\text{SO}_2$  in the gas, derived from the ferrocyanide and sulfuric acid reaction. The presence of sulfur was proved in the coatings, which consisted of zinc sulfide and oxide. The purification system was then modified by the addition of concentrated potassium hydrate solution between the  $\text{CO}$  generator and the gas storage, and the insertion of  $\text{N}/20$  iodine solution between the water wash and the phosphorus bottle; also when pure  $\text{CO}$  was being used, by the further addition of  $\text{KOH}$  solution between the phosphorus and the conc.  $\text{H}_2\text{SO}_4$  bottle.



The difficulty still persisted, and coatings containing sulfur were formed. Further investigation showed that very minute quantities of sulfuric anhydride mist, originating in the  $\text{CO}$  generator, passed unaltered through the long purification train and attacked the zinc. The ferrocyanide and sulfuric acid method of making carbon monoxide was therefore abandoned, and the method of passing  $\text{CO}_2$  over charcoal heated in an electric tube furnace was adopted. The  $\text{CO}_2$ , made from marble and dilute hydrochloric acid, was passed through  $\text{Na}_2\text{CO}_3$  solution, over the heated charcoal, through pyrogallic solution, and concentrated  $\text{KOH}$  solution, to the storage bottle. From the storage it passed through phosphorus, iodine solution, water, conc.  $\text{KOH}$ , (in case pure  $\text{CO}$  was being used), then through anhydrous calcium chloride, and phosphorus

pentoxide. The white zinc oxide deposit still formed, but no sulfur was now present. The formation of oxide was due to minute quantities of oxygen which were not removed by the pyrogallie solution and phosphorus, although the phosphorus was active, no inhibiting catalyzers being present, and the gas stream did not exceed 30 to 40 c.c. per minute.

The final purification train then adopted was as last described, with the addition of copper gauze and shredded copper placed in the glass tube, near the end where the gas stream entered, and the replacement of the iodine solution by  $\text{Na}_2\text{CO}_3$  solution. This train removed the troublesome foreign constituents from the gas stream, and further experiments gave consistent results.

The difficulties encountered are briefly outlined above for the purpose of showing how readily errors can be made in the investigation of a metal like zinc, which has such strong chemical affinity at the temperature of condensation.

For determining the composition of the gas mixture, the following method was used. The pure gas, or the mixture, would be accumulated in the storage bottle, which was then thoroughly shaken to insure a uniform gas. The copper was then heated to redness, and the gas was passed through the train at the rate of 30 to 40 c.c. per minute, until from 2500 to 3000 c.c. had been used. The sample was then taken in the manner described on page 283, an accurate glass stopcock burette, graduated to 0.1 c.c., being used in connection with Hempel gas pipettes for analysis. The analysis thus obtained represented the composition of the gas in contact with the zinc.<sup>21</sup> The gas burners under the zinc were then lighted and the experiment carried through to the end. Aside from CO and  $\text{CO}_2$ , the gas contained usually between 5 and 8 per cent. of nitrogen<sup>22</sup> and fractions of 1 per cent. of  $\text{H}_2$ . The source of the nitrogen is the charcoal, small amounts probably being due to the marble. The hydrogen comes from decomposed water vapor. The presence of these gases is not detrimental, since they represent normal constituents of distillation gases, which do not affect condensation.

### *Conclusions from Equilibrium Investigation*

Table 2 contains the experimental data and Fig. 5 is the equilibrium curve drawn from these data. The gas composition is recalculated on the basis of a mixture consisting of CO and  $\text{CO}_2$  only, omitting the nitrogen and hydrogen content. The results may be summed up in the statement that, within the condensation range between  $500^\circ$  and  $700^\circ \text{C.}$ , distillation gases must not contain more than 2.5 per cent.  $\text{CO}_2$  if oxidation of the zinc is to be avoided entirely; when present between 2.5 and 5 per cent.

<sup>21</sup> Metallic copper at red heat is without action on carbon dioxide or carbon monoxide. F. V. Bacho: *Monatsh* (1916) **37**, 119.

<sup>22</sup> Probably all nitrogen, though other gases related to  $\text{N}_2$  may be present.

TABLE 2.—*The Action of CO<sub>2</sub> on Zinc*

Number	Gas Composition before Passage		Gas Composition after Passage		Temperature °C.	Action on Zinc	Remarks
	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO			
26	0.8	99.2	....	.....	653	None	Zinc all bright. Slight carbon deposit on zinc in boat and on porcelain boat.
27	13.1	86.9	....	.....	635	Decided action	ZnO coat on tube. Typical blue powder formed. Zinc coated gray. ZnO crystals present.
28	2.3	97.7	1.7*	98.3	647	None	Zinc all bright. Carbon deposit evident, but slight.
29	5.6	94.4	5.6	94.4	533	Action present	Slight amount of oxide at head of boat. Zinc beyond bright.
29a	5.6	94.4	.....	.....	711	Decided action	ZnO formed at head and also further on; appears yellow when hot. Zinc in boat gray coated. ZnO crystals.
30	7.4	92.6	.....	.....	535	Distinct action	ZnO at head of boat. Carbon deposition noticeable. Carbon spots on zinc in boat. Color films on zinc.
31	4.5† 4.0	95.4† 96.0	2.1†	97.9†	534	Action slight	Slight amount of ZnO at head. Color films on zinc in boat. Some carbon deposit.
32	2.0† 2.2	98.0† 97.8	.....	.....	574	No action	Zinc all bright. Mirror. Carbon deposit slight.
32a	2.0	98.0	.....	.....	705	No action	Zinc bright.

\* Not certain; probably low.

† The upper figure is the composition of the gas after passing the heated tube containing copper; the lower figure is the composition before entering the tube. The difference may be considered as due to the decomposition of CO according to the reaction  $2\text{CO} \rightleftharpoons \text{CO}_2 + \text{C}$ , which is evidently not pronounced.

‡ Taken after standing in contact for some time, with no gas flow.

the action of CO<sub>2</sub> is comparatively slight; when more than 5 per cent. is present, oxidation is pronounced, and blue powder begins to form.

That decomposition of CO occurs in the condenser tube is evident from the slight carbon deposit formed, but it is small and its detection by analysis of the gas going in and coming out is uncertain, as the slight differences in composition are not readily determined. This confirms the data obtained from the experiments on composition of distillation gases, previously described, based on experiments 7 and 8. It is therefore concluded that in a condenser system in which there is a movement of gas not under normal speed (*i.e.*, one at which condensation of zinc vapor can readily take place) the decomposition of CO is not sufficient to interfere with condensation.

The reaction  $2\text{CO} \rightleftharpoons \text{CO}_2 + \text{C}$  has been thoroughly investigated,<sup>23</sup> and it need only be stated here that it proceeds from left to right much more rapidly at 500° than at 700°, and that the catalytic agents iron or nickel have a great influence on the speed of the reaction. It is advisable, therefore, in the design of a condenser, not to use structural material containing iron, or iron itself, in its interior; neither should the gas velocity be too slow, nor should the condenser be held at too low a temperature (450° to 500° C.). The last difficulty may be overcome by the use of a primary and a secondary condenser, the first being larger and held at the higher temperature range, while the second is smaller and held at the lower temperature range.

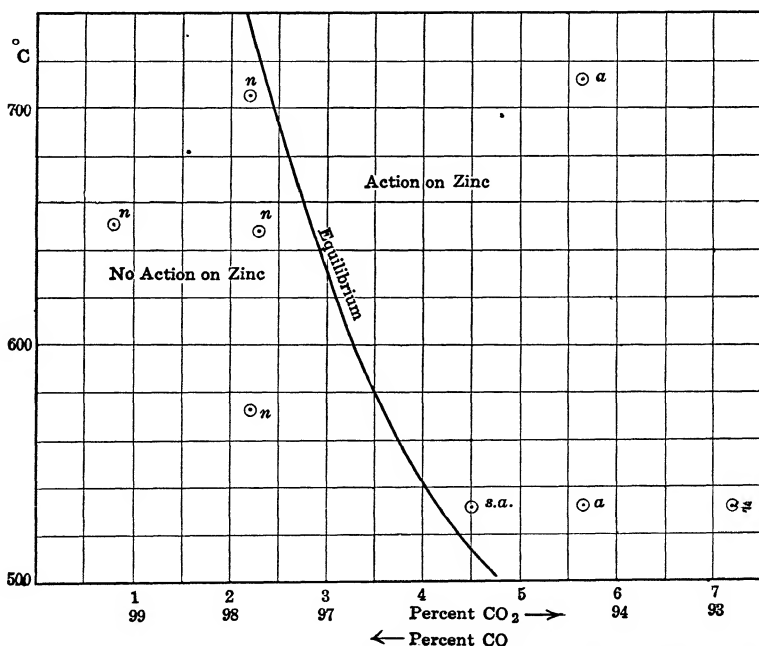


FIG. 5.—EQUILIBRIUM CURVE OF THE REACTION:  $\text{Zn} + \text{CO}_2 \rightleftharpoons \text{ZnO} + \text{CO}$ .

#### *Conditions of Zinc in Equilibrium Experiments*

In the preceding experiment, a relatively small amount of zinc vapor was contained in a large volume of gases. When the CO<sub>2</sub> was below the quantity necessary for attack on the zinc, the metal condensed beyond the heated zone of the tube in minute drops and globules firmly adherent to the glass, and all were mirror bright. Further along, a metal mirror would appear. In most of the condensing region the temperature was

<sup>23</sup> R. Schenck and W. Heller: *Berichte der Deutschen Chemische Gesellschaft* (1905) **38**, 2139; R. Schenck and F. Zimmermann: *Ibid* (1903) **36**, 1232.

below the melting point of zinc. The general appearance was very similar to that of water vapor condensing on a pane of glass. There was no loosely adherent powder, but toward the very end of the tube there were minute shining crystals of zinc. The zinc in the boat was partly bright and in part covered with a brown film and dark brown spots which were taken to be carbon.

When  $\text{CO}_2$  was present in sufficient amount to attack the zinc readily, loosely adherent gray coatings and deposits formed above the boat, and further along, typical blue powder, accompanied by bright metal globules and white oxide films.

In the absence of interfering substances, the condensation of zinc vapor into liquid metal drops, and the coalescence of these drops into a bath of metal, present no particular difficulties. The coalescence of the zinc drops is governed by surface tension and related forces, and if these could be diminished, condensing surfaces might be made more efficient. The subject presents an interesting field for investigation.

#### REQUISITES FOR SUCCESSFUL CONDENSATION

The object of the experimental work detailed in this paper was to determine certain fundamental facts in the distillation and condensation of zinc, in order to find an answer to the question—Are any products formed during the distillation of zinc ore and the subsequent condensation of the zinc vapor which interfere with the condensation to liquid metal; if so, what are they, and in what proportion are they harmful?

The results of the investigation show that under proper conditions there is nothing to interfere with the condensation of the zinc to liquid metal, but that the latitude within which operations may be carried on is not wide. One requisite is that the distillation and condensation systems must be tight and impervious to air. This, of course, has been well known, but the fundamental reason for it has not been recognized. Many of the electric zinc furnaces that have been experimented with, or proposed, suffer from this vital defect, the arc furnace being particularly defective on this point. If operated continuously, by feeding ore and reduction carbon, and discharging slag, there will be regions in the furnace, in the cooler portions above the smelting arcs, where carbon dioxide will form in considerable proportion, according to the principles discussed in this paper. For an electric zinc smelting furnace to be successful, it must be able to obtain a uniform temperature rapidly throughout its whole distillation chamber; certainly no part of the chamber or its contents should be below  $950^\circ$  to  $1000^\circ \text{C}$ . From the standpoint of the condensation of vapor, it is not possible to distil at  $1200^\circ \text{C}$ . in one part of the chamber, while the products of distillation come in contact with ore charge at below  $1000^\circ \text{C}$ . in another part, and at the same time condense a large proportion of the vapor to liquid spelter.



## DISCUSSION

E. E. THUM,\* Salt Lake City, Utah. (Written discussion†).—Dr. Fulton's paper sheds a great deal of light upon questions which have perplexed many who have studied the metallurgy of zinc, and have fruitlessly brought forth several hypotheses and reams of discussion. It would be a bright day if others would follow Dr. Fulton's example, for, as a prominent metallurgist recently said, the zinc smelters' adherence to old practices is not so much for the reason that no research has been undertaken, for a great deal has been, but rather because the results have not been published.

On page 288, Dr. Fulton states that carbon dioxide in excess of 1 per cent. is not found in the gases distilled above the carbon-reduction temperature; yet in Table 1, page 284, we find in experiment 8b, at perhaps 1500° C., that CO<sub>2</sub> is given as 2.5 per cent. Other results, notably experiment 12c at 1500° C., show higher percentages of carbon dioxide. Dr. Fulton refers to this 1 per cent. carbon dioxide maximum several times later in the paper, apparently at some discrepancy with his experimental results.

On page 298, Dr. Fulton states that the reaction  $2\text{CO} \rightleftharpoons \text{CO}_2 + \text{C}$  proceeds from left to right much more rapidly at 500° than at 700°. That is true, of course, since CO<sub>2</sub> is the usual form at low temperature; yet on page 288, in discussing the effect of carbon dioxide on the condensation, he states that at the end of the distillation the proportion of dioxide increases, particularly when the temperature is high (above 1400°), the monoxide suffering decomposition into carbon dioxide and carbon. That is a point, it seems to me, that Dr. Fulton would do well to elucidate at some length. The reaction he indicates is, of course, a reversible reaction, and the equilibrium concentration-temperature diagram has been well worked out, showing that carbon dioxide is stable at low temperature, but reverts to carbon monoxide at higher temperatures. Now, if that reaction again reverses at 1000° C. and higher, it would be a rather peculiar reaction and of a type which is not at all common in physical chemistry, since highest-temperature decomposition of carbon monoxide might produce carbon and oxygen rather than carbon dioxide and carbon.

Dr. Fulton says that his experiments were conducted under rigorous conditions to determine the gas from the distillation chamber. He states that "For this purpose, porcelain and quartz tubes were used, having been demonstrated to be tight and refractory enough for the temperatures employed." These at times reached 1500° C. One who is familiar with the rapid deterioration of platinum thermocouples in

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\* Western Editor, *Chemical and Metallurgical Engineering*.

† Received Sept. 28, 1918.

reducing atmospheres at 1500° C., even when protected by the best fused-quartz tubes, would doubtless be glad to be reassured as to this point. Doubtless Dr. Fulton will give us detailed information as to the tests he made to establish the imperviousness of his tubing, especially since Alleman and Darlington, of Swarthmore College, were forced to great extremes before they were able to construct a furnace that was gas tight at such high temperatures.<sup>1</sup> These two scientists ascribe much of the variation in published results on occluded gases in metals to insufficient precautions to insure against leakage of gases through the walls of the apparatus in use.

CHARLES H. FULTON (author's reply to discussion\*).—Mr. Thum is kind enough to point out an apparent discrepancy between the conclusions drawn from experiments and the results of those experiments. It is stated on page 288 that carbon dioxide in excess of 1 per cent. is not found in the gases above the carbon reduction temperature (of zinc oxide). This statement refers to such temperatures as occur in ordinary zinc distillation and is not meant to include temperatures above 1350° C., and it should have been so stated in the original paper. When the temperature of the distilling chamber reached 1400° and above, it was noted that the amount of carbon dioxide in the gases increased. This cannot be explained by the equilibrium of the reaction  $2\text{CO} \rightleftharpoons \text{CO}_2 + \text{C}$ , and in seeking reasons for the discrepancy, the experiments of Meyer and Langer, cited in the original paper, were found. These experiments were carried out for the purpose of studying decomposition of gases at high temperature and the determination of molecular weights. The temperatures were attained in a platinum tube, proved tight by experiment. The paper states that at 1200° C. carbon monoxide remains unchanged, but that at 1690° C. there was a partial decomposition into carbon dioxide and carbon. Carbon dioxide at this high temperature showed traces only of decomposition. According to Nernst and von Wartenberg, the decomposition is into carbon monoxide and oxygen.<sup>2</sup>

These results offer an explanation for the increase of carbon dioxide in the distillation gases produced at high temperatures. The analysis of the gas, of course, is made after it has passed from the point of origin in the high temperature region, through the region of intermediate temperature, to the point of sampling. What happens in the interval between origin and sampling is not known, and the gas analysis reveals only the final result. It is perfectly possible that instead of  $2\text{CO} \rightleftharpoons$

<sup>1</sup> G. Alleman and C. J. Darlington: Occluded Gases in Ferrous Alloys. *Journal, Franklin Inst.* (Feb., 1918) **185**, 161, 333, 461.

\* Received Nov. 29, 1918.

<sup>2</sup> W. Nernst and H. von Wartenberg: Die Dissociation von Wasserdampf. *Zeitschrift für Physicalische Chemie* (1906) **56**, 534.

$\text{CO}_2 + \text{C}$  reversing itself at the high temperature and proceeding from left to right, the carbon monoxide is partly dissociated into carbon and oxygen and then that, as the gas passes to cooler regions, the oxygen unites with carbon monoxide with the consequent production of carbon dioxide. The fact remains that when the temperature of distillation is high at the end increased quantities of carbon dioxide appear.

The tightness of the quartz and porcelain tubes used was tested as follows: The middle portion of the tube was heated to a maximum normal distilling temperature of  $1350^\circ \text{C}$ . in the electric furnace, the atmosphere around the outside of the tubes being essentially one of carbon monoxide due to the nature of the electric furnace. Purified nitrogen gas was then passed through the tube; this was carefully analyzed before entrance and after passage through the tubes, and no change in gas composition was noted. Whether the tubes are tight at  $1500^\circ \text{C}$ ., I am unable to say. The high temperatures of  $1400$  to  $1500^\circ \text{C}$ . were not the normal temperatures worked with and were reached only occasionally at the very end of the distillation and then prevailed for very short periods of time. The use of thermocouples for the high temperatures, in reducing atmospheres, particularly when zinc vapor is present, presents serious difficulties. In the highest temperatures measured in the experimental work detailed in the paper, the hot junction end of the couple was in each case destroyed and the couple had to be shortened and recalibrated before it could be used again.

## Effect of Impurities on Hardness of Cast Zinc or Spelter\*

BY G. C. STONE,† NEW YORK, N. Y.

(Milwaukee Meeting, October, 1919)

As the term "hardness" is used with many different meanings, it should be understood that it here means resistance to deformation by compression. It was determined by measuring the pressure, in pounds per square inch, necessary to reduce by 20 per cent., the length of cylinders 1 in. (25 mm.) in diameter by 2.6 in. (66 mm.) long. The tests were made at Columbia University on an Olsen 400,000-lb. machine, the load being applied at a constant rate of speed 0.0319 in. per min. The results were obtained by Messrs. Rigg and Williams, and some of them were given in a paper read at the Sixteenth Annual Meeting of the American Society for Testing Materials, in June, 1913. The remainder of the results were obtained in subsequent experiments by the same authors. In all cases, the results given are the averages of three or more closely concordant tests.

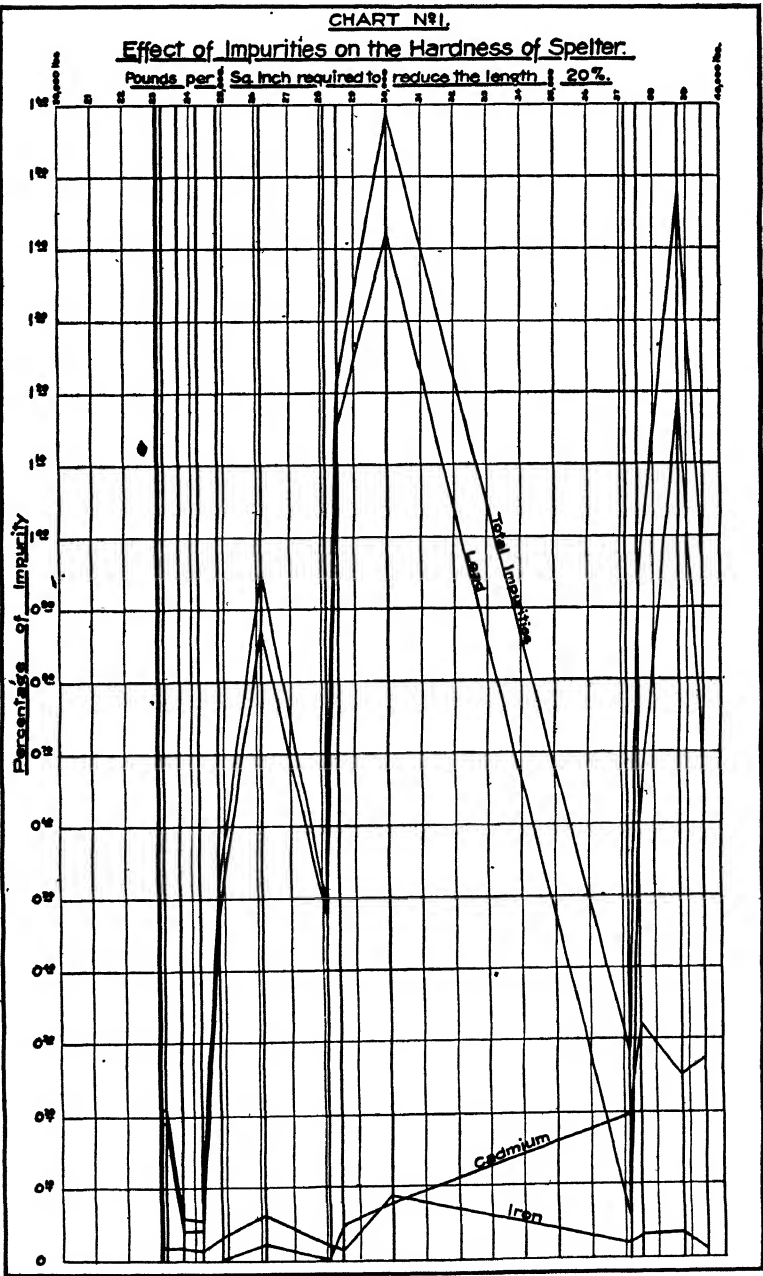
All of the spelters tested were of standard commercial grades and all were of compositions making them good deliveries. The accompanying table gives the results arranged in the order of increasing hardness:

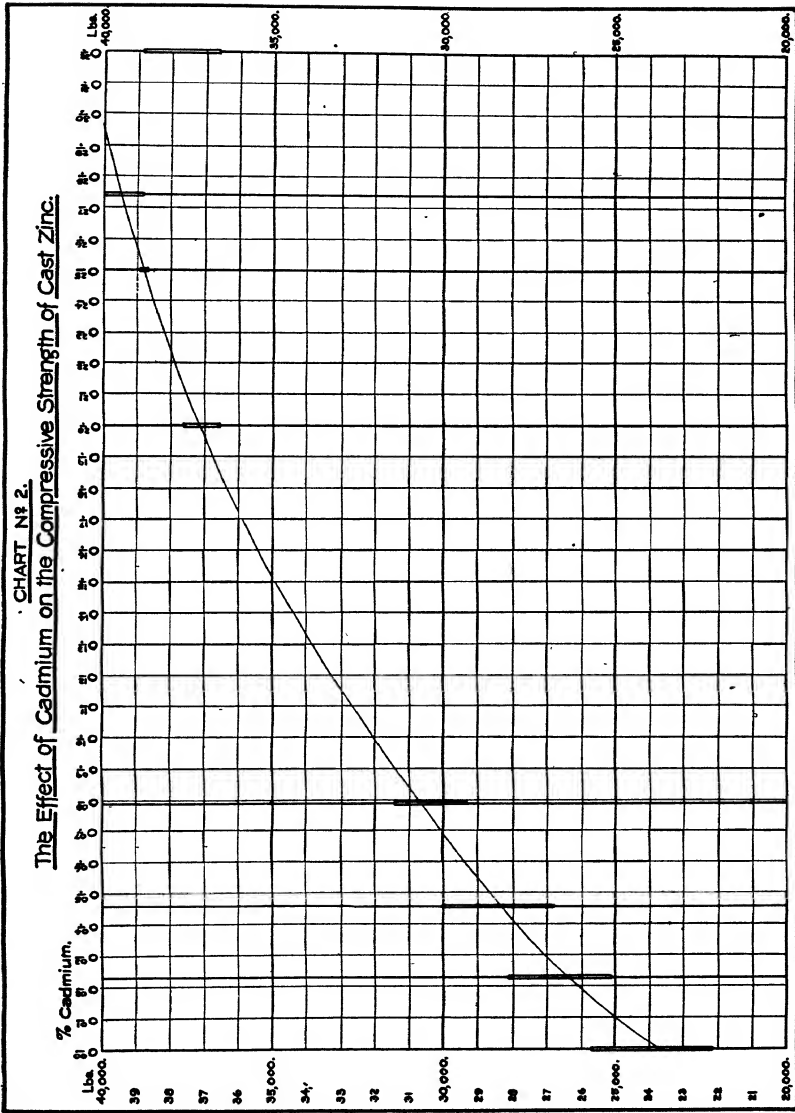
TABLE 1.—*Hardness of Commercial Spelters*

No.	Lead	Iron	Cadmium	Total Impurities, Per Cent.	Lb. per Sq. Inch for 20 Per Cent.
1	0 194	0 016	0.000	0.210	23,030
2	0.190	0.017	0.000	0.207	23,150
3	0.040	0.016	0.000	0.056	23,640
4	0.041	0.014	0.000	0.055	24,230
5	0 484	0 031	0.000	0.515	24,830
6	0.870	0 062	0.023	0.955	26,140
7	0 474	0.017	0.000	0.491	28,070
8	1.150	0.011	0.046	1.207	28,450
9	1.420	0.087	0.079	1.586	29,970
10	0.060	0.019	0.200	0.279	37,170
11	0.600	0.030	0.320	0.950	37,540
12	1.190	0 032	0 250	1.472	38,770
13	0.680	0.010	0.274	0.964	39,490

\* This paper was first printed in the *Journal* of the American Institute of Metals, March, 1918.

† New Jersey Zinc Co.





The results are plotted in Chart 1. An examination of either the table or chart shows no indications of a definite relation between the lead, iron or total impurities and the hardness. Both show that increased cadmium gives greater hardness.

To test the closeness of this relationship, Chart 2 was drawn. In this, the percentage of cadmium forms the abscissas and the pressure the ordinates. The maximum and minimum limits of compression are shown, and a curve can be drawn well within these limits; in fact, it is close to the average in all cases. It seems obvious that cadmium is practically the sole cause of hardness in commercial spelter. Lead alone has practically no effect.

Iron up to 0.10 per cent. (the extreme limit for salable spelter) has no effect. Iron in large quantities, 2 to 3 per cent., makes a very hard and brittle alloy, but it is dross and not spelter. The hardening and consequent brittleness, due to the presence of cadmium, would account for the fact that American rolling mills usually have more trouble and loss from cracked edges than European mills, as the American ores are usually higher in cadmium than the European.

## Oxygen and Sulfur in the Melting of Copper Cathodes

BY STANISLAUS SKOWRONSKI,\* B. S., PERTH AMBOY, N. J.

(Colorado Meeting, September, 1918)

THE melting of cathode copper, usually containing 99.98+ per cent. Cu, would appear to be a simple matter. Owing to the well known affinity of copper for sulfur, however, so much sulfur is absorbed by the copper during the operation that a long and expensive refining process is required to remove it, and the final product is inferior in purity and lower in electrical conductivity than the original cathode copper.

The furnace operation, as now conducted, includes the following stages:

1. *Charging* the cathodes into the furnace.

2. *Melting* the cathodes.

3. *Oxidizing period*—saturating the copper with cuprous sub-oxide and bringing the metal to the condition known as "set copper;" this is now done by blowing air into the molten bath of metal.

4. *Reduction period*, otherwise known as "poling," whereby the oxygen previously added is removed by covering the molten bath completely with coke or charcoal and introducing large poles of green wood; this reduces the cuprous sub-oxide, bringing the copper to the "tough pitch" stage ready for casting.

5. *Casting Period*.

Many experiments have been made to obviate the long periods of oxidation and subsequent reduction, with no success. While the copper first cast will be good, as indicated by the set surface of the bars, it is impossible to hold it to the "tough pitch" condition, and long before the whole charge has been poured the copper will become "overpoled," entailing numerous difficulties.

Sulfur has always been blamed by the furnace men as the reason why this refining process is necessary, yet the literature on the subject does not sufficiently emphasize how small a proportion of sulfur will ruin copper for practical purposes.

The sulfur absorbed by copper during the melting period comes from two sources: (1) the sulfate left on the cathode sheets in the tank houses; (2) the fuel. The percentage of sulfates on the cathodes is variable, and depends on the care taken in washing the cathodes after removing

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\* Head of Research Dept., Raritan Copper Works.



them from the electrolytic tanks. Crystallized copper sulfate can often be seen on the top edges, particularly around the loops of the cathodes, and even well washed cathodes will show from 0.002 to 0.004 per cent. of sulfur. In the furnace, the copper sulfate becomes dehydrated and considerable quantities of it are blown out of the furnace, most of this being caught in the flues of the waste-heat boilers; samples of flue dust collected beyond these boilers analyzed 16.5 per cent. of water-soluble sulfates. The fuel, whether soft coal or oil, always contains an appreciable amount of sulfur, which is likely to be absorbed by the copper, as the products of combustion pass over the charge.

## MELTING UNDER COKE COVER

In order to study the behavior of sulfur and oxygen during the refining of cathode copper, samples were taken at  $\frac{1}{2}$ -hr. intervals from

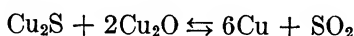
TABLE 1.—*Oxygen and Sulfur Contents of Copper at Various Stages of Refining, under Coke Cover*

Sample	Time	Period	Total Time in Furnace, Hours	Per Cent. Oxygen	Per Cent. Sulfur	Conductivity Annealed Wire
	5:00 p. m.	Charging				
	7:10 p. m.	Finished charging				
1	5:00 a. m.	Started blowing air	12	0.170	0.0030	98.6
2	5:30 a. m.	Oxidizing. . . . .		0.194	0.0035	
3	6:00 a. m.	Oxidizing. . . . .	13	0.203	0.0026	
4	6:30 a. m.	Oxidizing. . . . .	..	0.240	0.0015	Wire
5	7:00 a. m.	Oxidizing. . . . .	14	0.277	0.0010	could
6	7:30 a. m.	Oxidizing. . . . .	..	0.275	0.0007	not be
7	8:00 a. m.	Oxidizing. . . . .	15	0.293	0.0005	cold
8	8:30 a. m.	Oxidizing. . . . .	..	0.302	0.0003	drawn
9	9:00 a. m.	Oxidizing. . . . .	16	0.323	Nil	owing to
10	9:30 a. m.	Oxidizing. . . . .	..	0.337	Nil	high oxy-
11	10:00 a. m.	Oxidizing. . . . .	17	0.366	Nil	gen con-
12	10:30 a. m.	Oxidizing. . . . .	..	0.410	Nil	tents
	10:45 a. m.	Furnace coked. . . . .	..	..	..	
13	11:00 a. m.	Poling. . . . .	18	0.335	Nil	
14	11:30 a. m.	Poling. . . . .	..	0.214	Nil	
15	12:00 m.	Poling. . . . .	19	0.108	Nil	99.8
16	12:30 p. m.	Poling. . . . .	..	0.073	0.0009	100.0
17	1:00 p. m.	Poling. . . . .	20	0.045	0.0014	100.2
	1:30 p. m.	Casting				
18	2:00 p. m.	Casting. . . . .	21	0.048	0.0014	99.9
19	3:00 p. m.	Casting. . . . .	22	0.039	0.0029	100.1
20	4:00 p. m.	Casting. . . . .	23	0.048	0.0031	99.9
21	5:00 p. m.	Casting. . . . .	24	0.051	0.0019	99.9
	5.15 p. m.	Charge out. . . . .	24 $\frac{1}{4}$			

a 500,000-lb. charge of copper. Results are compiled in Table 1 and shown graphically in Fig. 1.

Analysis of the final product gave 99.93 per cent. Cu, and the average conductivity of the samples was 100.

Referring to Fig. 1, during the oxidizing period, as expected, the oxygen percentage steadily increases while the sulfur continuously decreases, until the reversible reaction



has been completed so far as the cuprous sulfide in the copper is concerned. Further, no sulfur is absorbed, that is, the reaction does not

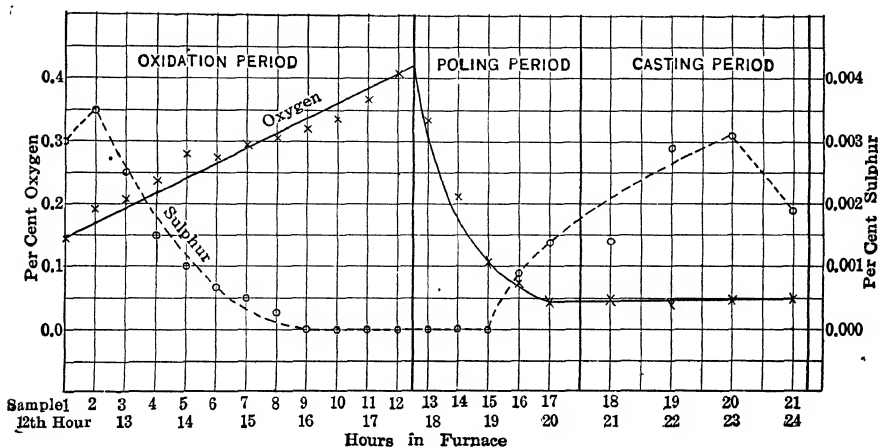


FIG. 1.—BEHAVIOR OF SULFUR AND OXYGEN DURING REFINING UNDER COKE COVER.

become reversible until the charge has been brought up to nearly the "tough pitch" stage, when it again takes up sulfur.

This residual sulfur, amounting to only 15 lb. in the whole charge, can come from two sources only: the fuel and the coke used to cover the charge. A good grade of 72-hr. coke, analyzing 0.83 per cent. S, was used, and as 1200 lb. was required, this introduced 10 lb. of sulfur into the charge.

#### MELTING UNDER CHARCOAL COVER

This test was duplicated on another furnace charge of the same weight, using charcoal instead of coke to cover the charge. This charcoal contained only 0.03 per cent. S. The results are compiled in Table 2 and shown graphically in Fig. 2.

The analysis of the final product gave 99.966 per cent. Cu, and the average conductivity of the samples was 100.5

TABLE 2.—Oxygen and Sulfur Contents of Copper at Various Stages of Refining, under Charcoal Cover

Sample	Time	Period	Total Time in Furnace, Hours	Per Cent. Oxygen	Per Cent. Sulfur	Conductivity Annealed Wire
	7:30 p. m.	Charging.....				
	9:10 p. m.	Finished charging...				
1	10:00 a. m.	Oxidizing.....	14½	0.249	0.0020	
2	11:00 a. m.	Oxidizing.....		0.380	0.0009	
3	12:00 p. m.	Oxidizing.....		0.387	0.0004	
4	1:00 p. m.	Oxidizing.....		0.399	Nil	
5	2:00 p. m.	Oxidizing.....		0.447	Nil	
6	3:00 p. m.	Oxidizing.....		0.467	Nil	
7	3:30 p. m.	Poling.....	20	0.472	Nil	
8	4:00 p. m.	Poling.....		0.470	Nil	
9	5:00 p. m.	Poling.....		0.221	Nil	
10	6:00 p. m.	Poling.....		0.059	Nil	100.1
11	6:30 p. m.	Casting.....	23	0.019	0.0010	100.6
12	7:30 p. m.	Casting.....		0.018	0.0013	100.7
13	8:30 p. m.	Casting.....		0.020	0.0011	100.4
14	9:30 p. m.	Casting.....		0.024	0.0010	100.4
15	10:30 p. m.	Casting.....	27	0.021	0.0011	100.4
	10:40 p. m.	Charge out.....				

Referring to Fig. 2, the behavior of oxygen and sulfur during refining follows closely that of the former charge; but owing to the absence of

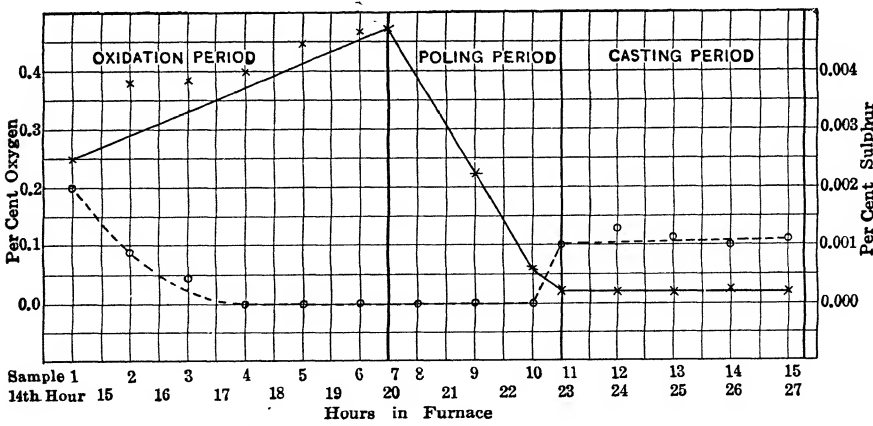


FIG. 2.—BEHAVIOR OF SULFUR AND OXYGEN DURING REFINING UNDER CHARCOAL COVER.

sulfur in the charcoal used to cover the charge during the poling and casting periods, the absorption of sulfur by the copper was much less.

TABLE 3.—*Comparison of Copper Refined under Coke and under Charcoal*

	1. COKE- COVERED CHARGE	2. CHARCOAL- COVERED CHARGE
Sulfur, average per cent. . . . .	0.0024	0.0011
Oxygen, average per cent. . . . .	0.045	0.021
Copper, average per cent. . . . .	99.937	99.966
Conductivity. . . . .	100.0	100.5
Sulfur in charge, pounds. . . . .	15.0	5.5

The comparison of the two charges, given in Table 3, shows not only a lower sulfur content for the charge refined under charcoal, but also lower oxygen contents, and a better quality of copper.

It is becoming better understood that the "surface set," or the pitch of copper, is directly controlled by the sulfur and perhaps certain reducing gases, such as carbon monoxide and hydrogen, rather than by oxygen; and that sulfur raises the "set" or overpoles the copper, thus necessitating a certain amount of oxygen, which depresses the "set", to counterbalance the action of the sulfur.

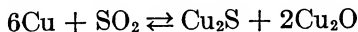
In these two charges, the "pitch" of the copper was the same, yet owing to the lower sulfur in product No. 2, its oxygen contents is also much lower than that of No. 1, affording great improvement in the quality of the copper. The refiner did not know how much sulfur was present in his final product; he simply worked the copper to the desired pitch, and the sulfur present in each charge determined the amount of oxygen necessary to give the proper "surface set" on the copper.

#### SUMMARY

Samples taken at regular intervals during the melting and refining of copper cathodes show:

1. The total elimination of sulfur during the "oxidation" period and before poling.

2. The re-absorption of sulfur by the charge when the percentage of cuprous oxide is low enough to allow the reversible reaction



to take place.

3. The advisability and importance of using a low-sulfur coke or charcoal to cover the molten bath during the poling and casting periods.

### Relation of Sulfur to the Overpoling of Copper

BY STANISLAUS SKOWRONSKI,\* B. S., PERTH AMBOY, N. J.

(Colorado Meeting, September, 1918)

OVERPOLED copper, as commonly defined, is copper which has been excessively reduced during the poling period of the refining process. Owing to its porosity, such copper is unfit for commercial purposes.

The cause of this phenomenon is undoubtedly the lack of oxides in the copper, yet this does not completely explain it. Refined copper of the same "pitch" and "set" varies considerably in oxygen contents, and analyses of overpoled bars also show similar wide differences; moreover, it is possible to melt copper in crucibles so as to obtain bars that are practically free from oxygen although they show a "tough pitch" and "level set" surface.

Frederick Johnson advances the following theory:

The regulation of the quantity of oxygen in copper is the keynote of all copper refining. It is the presence of a well defined proportion of oxygen in the form of cuprous oxide, forming a copper-cuprous oxide eutectic, which enables copper to be cast free from blowholes. It seems that when oxygen is present, other gases are less soluble in molten copper; when oxygen is absent, or is present in insufficient quantity, the copper dissolves other gases, such as hydrogen and carbon monoxide, which are insoluble in the copper when solid, and, being rejected during the process of solidification, causes internal porosity and external ridges and excrescences.<sup>1</sup>

\* \* \* \* \*

The "level set" of wire bars, etc., is entirely the result of a compromise between the occluded gases and cuprous oxide. With excess of the former the metal would be "overpitch" and spongy, while an excess of the latter would result in the metal being "underpitch" and brittle.<sup>2</sup>

The result of the absorption and subsequent expulsion of reducing gases, carbon monoxide and hydrogen, and the effect of this action on the overpoling of copper are in dispute; the literature on the subject is full of contradictory statements and will continue to be so until better analytical methods have been developed. Sulfur, however, is tangible. The refiner can see it in his button sample and consequently attributes to it any trouble that may happen during the melting of the copper.

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\* Head of Research Dept., Raritan Copper Works.

<sup>1</sup> *Journal*, Institute of Metals (No. 2, 1910) 4, 165.

<sup>2</sup> *Journal*, Society of Chemical Industry (July 31, 1917) 36, 804.

The cathode copper used in the following experiments was of high purity, analyzing 99.99 per cent. Cu. In all melts, 1500 gm. of copper were used, making a standard-size test bar. The graphite crucibles were thoroughly annealed and burned before using. The furnace was a small, gas-fired, crucible furnace. Conductivity measurements were made on all test bars and are reported in terms of the Matthiessen standard, soft annealed wire.

### MELTING CATHODE COPPER

To prevent oxidation, cathode copper was melted under a flux of soda-ash, and also under charcoal, with results shown in Table 1.

TABLE 1.—*Melting Cathode Copper*

	Before Melting	After Melting	
		Under Soda-Ash	Under Charcoal
Copper. ....	99.990	99.986	99.960
Oxygen. ....	Nil	0.003	0.029
Sulfur. ....	Nil	Nil	Nil
Conductivity. ....	101.9	101.3	100.8

All test bars made in these experiments had a perfect "level set" surface. In duplicate tests, attempts were made to overpole the copper by poling with green wood, but copper could not be thus overpoled, all test bars so made having "level" set and proper "pitch."

It is evident from these results that, since it is possible to obtain "tough pitch" or "level set" copper without oxygen, the overpoling of copper cannot be considered as due to a lack of oxygen; on the contrary, the results tend to prove Johnson's theory that another factor, which the oxygen is only counteracting, must be taken into consideration.

### SULFUR ALLOYS

The method of making sulfur alloys was to place a weighed amount of cuprous sulfide (20.12 per cent. S.) in the bottom of a graphite crucible and pile the charge of sheared copper cathodes on top of it. Although the cuprous sulfide was added before melting, little sulfur escaped, most of it alloying with the copper. Results are shown in Table 2.

Bars No. 2 and 3A were melted under charcoal, while bars No. 3B, 4, 5 and 6 were not. It is to be noted that while test bars 3A and 3B contained the same amount of sulfur, bar 3A was overpoled with 0.022 per cent. of oxygen while bar 3B, containing 0.071 per cent. of

TABLE 2.—*Addition of Sulfur to Copper*

No.	Sulfur, Per Cent.	Oxygen, Per Cent.	Conductivity	Set Surface on Test Bars
2	0.0012	0.023	100.7	Level, O. K.
3A	0.0038	0.022	100.1	Overpoled
3B	0.0038	0.071	99.5	Level, O. K.
4	0.0052	0.083	98.5	Badly overpoled and spewed
5	0.0058	0.093	98.8	
6	0.0069	0.072	98.6	

oxygen, was not; yet bars No. 4, 5 and 6, all high in sulfur, were badly overpoled with higher oxygen contents than bar No. 3B.

## EFFECT OF CHARCOAL COVER

Two series of experiments, varying the sulfur contents while keeping the oxygen as constant as possible, were made. In series 1, the crucibles were left uncovered and the copper allowed to absorb all the oxygen it could. In series 2 the copper was melted under charcoal and poled before casting. Comparative results are shown in Tables 3 and 4.

TABLE 3.—*Copper Melted in Uncovered Crucibles*

Bar No.	Cu <sub>2</sub> S Added, Grams	Calculated Per Cent. Sulfur	Analysis of Test Bars			
			Sulfur	Oxygen	Copper	Conductivity
1	0.10	0.0013	0.0009	0.079	99.902	99.8
2	0.20	0.0027	0.0021	0.056	99.928	100.2
3	0.30	0.0040	0.0033	0.113	99.874	99.5
4	0.40	0.0054	0.0044	0.097	99.886	99.3
5	0.50	0.0067	0.0039	0.127	99.846	98.9

Photographs of the bars referred to in Table 3 are shown in Fig. 1. Except No. 4, all bars had a "level set" surface, showing no evidence whatever of overpoling. Bar No. 4 had the characteristic ridge in its middle, showing a slightly overpoled condition of the copper.

Photographs of the bars referred to in Table 4 are shown in Fig. 2.

Bar No. 1 was strictly O. K.

Bar No. 2 had slight crown, slightly overpoled.

Bar No. 3 had ridge in the middle of bar and sulfur worms.

Bars No. 4 and 5 were decidedly overpoled and spewed badly.

From these results, it is evident that cuprous oxide counteracts the effect of sulfur, either by increasing the solubility of sulfur in copper, or, as pointed out by Johnson, by inducing a physical condition of equi-

TABLE 4.—*Copper Melted under Charcoal*

Bar No.	Cu <sub>2</sub> S Added, Grams	Calculated Per Cent. Sulfur	Analysis of Test Bars			
			Sulfur	Oxygen	Copper	Conductivity
1	0.10	0.0013	0.0008	0.022	99.954	100.7
2	0.20	0.0027	0.0015	0.016	99.962	101.0
3	0.30	0.0040	0.0028	0.016	99.956	100.8
4	0.40	0.0054	0.0042	0.025	99.960	100.3
5	0.50	0.0067	0.0045	0.034	99.958	100.5

librium between the sulfur, tending to “overpole” the copper, and the cuprous oxide, tending to “underpole” it. Yet neither theory explains

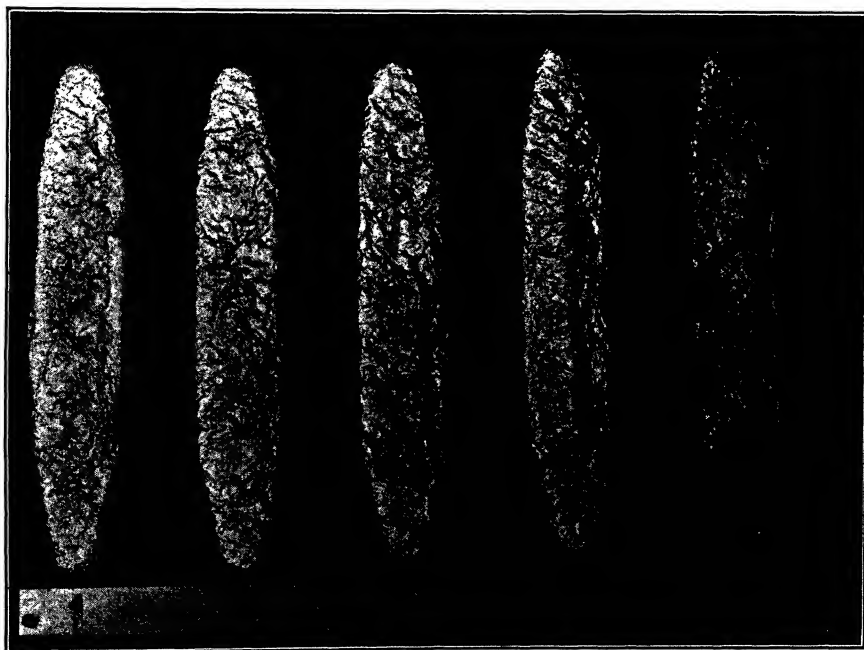


FIG. 1.—COPPER MELTED IN UNCOVERED CRUCIBLES.

the fact that when a charge of copper is “overpoled” it cannot be righted by a simple addition of cuprous oxide, but the charge must be reworked to set copper.

#### EFFECT OF SULFUR ON THE CONDUCTIVITY OF COPPER

A comparison of the conductivity tests arranged according to increasing sulfur contents is shown in Table 5.



TABLE 5.—*Effect of Sulfur on Conductivity*

	Per Cent. Sulfur	Per Cent. Oxygen	Conductivity
Cathode copper.....	Nil	Nil	101.9
Copper melted under flux.....	Nil	0.003	101.3
Copper melted under charcoal.....	Nil	0.029	100.8
	0.0008	0.022	100.7
	0.0009	0.079	99.8
	0.0012	0.023	100.7
	0.0015	0.016	101.0
	0.0021	0.056	100.2
	0.0028	0.016	100.8
	0.0033	0.113	99.5
	0.0038	0.022	100.1
Sulfur alloys.....	0.0038	0.071	99.5
	0.0039	0.127	98.9
	0.0042	0.025	100.3
	0.0044	0.097	99.3
	0.0045	0.034	100.5
	0.0052	0.083	98.5
	0.0058	0.093	98.8
	0.0069	0.072	98.6



FIG. 2.—COPPER MELTED UNDER CHARCOAL AND POLED BEFORE POURING.

Since the sulfur contents of refined copper is within the limits of 0.001 to 0.004 per cent., the conductivity of commercial copper cannot be said to be directly affected by the sulfur it contains. It is the cuprous oxide, which must be left in the copper to counteract the sulfur, that lowers the conductivity. This is more evident by comparing Tables 3 and 4, where the overpoled bars show high electrical conductivities, whereas the "level set" bars, high in oxygen, show low conductivity.

It takes a comparatively large amount of cuprous oxide to counteract a trace of sulfur; this explains why, in the refining of copper, the gases from fresh fuel will often "overpole" the copper during the casting period, and also why copper cannot be held in the furnace at the proper pitch for more than a few hours. It also explains the limit of recharging cathode sheets into the furnace after the copper has been worked to the proper pitch, the sulfate present on the cathode sheets overbalancing the equilibrium established between the cuprous oxide and the sulfur.

While cuprous sulfide was used in these tests, one series of experiments was made using dehydrated copper sulfate; the same results were obtained as to the physical characteristics and "set" of the copper, showing that sulfur will be absorbed by the copper whether present as sulfide or as sulfate.

#### SUMMARY

Experiments on alloys of sulfur and copper show that:

1. Sulfur is a direct agent in the overpoling of copper, the cuprous oxide left in the copper acting only as a neutralizing agent for the sulfur.

2. The amount of oxygen present in refined copper, as regulated by the "pitch" of the copper, is automatically controlled by the sulfur, and possibly by other reducing gases present in the copper. (No reliable data regarding the second statement are at hand.)

3. It is impossible to overpole copper by over-reduction if the copper does not contain sulfur and possibly other reducing gases.

Since no reliable data are at hand as to the actual effect of hydrogen and carbon monoxide on the overpoling of copper, the conclusion as to sulfur must be modified somewhat so as to include the possibility that these reducing gases may also exert some effect, although the overpoling of copper free from sulfur has not been found to be possible in these investigations.

#### DISCUSSION

G. H. CLAMER\* (Philadelphia, Pa.).—Sulfur is not so detrimental in its effect if oxygen is also present, so that in sulfur-carrying bronzes and brasses, it is advantageous to maintain a certain percentage of oxygen.

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\* First Vice-president and Secretary, Ajax Metal Co.

As a matter of fact, it would be possible sometimes to correct difficulties by actually adding oxygen. If the cathodes were charged directly into an electric furnace in a neutral or reducing atmosphere, a very high grade of copper would result by a simple melting operation. This would very largely eliminate the loss occasioned by melting in a reverberatory furnace. In such a furnace a large amount of slag is produced during the oxidizing stage of refining. Notwithstanding the fact that melting in an electric furnace might be somewhat more expensive than melting in a fuel furnace, the ultimate result would be a saving and the production of a very high grade of copper.

PHILIP L. GILL, New York, N. Y. (written discussion\*).—There is one feature of the fire-refining of electrolytic copper which I believe should be mentioned when the relation of oxygen content to the "pitch" or "set" of refined copper is under discussion; that is, the segregation of impurities. Variation of "pitch" or "set" is frequently noted in castings of different shapes, cast at the same time from the same furnace, though the oxygen content remains identical.

For instance, it is possible to cast two wire-bars in the same shaped molds from the same ladleful of copper, the one 2 in. thick showing a good level set, the other, 4 in. thick, being overpoled. Also, when two bars are cast of the same weight and at the same time from the same furnace, one bar in a copper mold at the proper temperature, the other in a mold that has been allowed to get overheated, the bar in the hot mold will show signs of higher "pitch" or "set." Again, the temperature at which the copper is cast enters into the set of the castings. A hot copper with a high-crowned set can be quickly converted to a flat level set by lowering the temperature of the metal in the furnace by addition of a few hundred pounds of high-pitch wire-bar.

Segregation can also be noted in the fact that bars cast by pouring the metal entirely on one end show a lower set at the point of pouring than on the opposite end. If copper cast in this manner becomes overpoled, the wire-bars will show indications of overpoling first on the end opposite to the one from which they have been poured.

The difficulty of making sound copper castings in closed green-sand molds, without the aid of heavy risers, is well known to many foundrymen, and is another indication of how difficult it is to maintain the balance between the neutralizing cuprous oxide and the reducing gases absorbed by molten copper when conditions in the casting are favorable to segregation.

Referring to his Table 1, the results of melting cathode copper in 1500-gm. lots in graphite crucibles under soda ash and charcoal covers, Mr. Skowronski states that it was impossible to overpole this copper by

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\* Received June 22, 1918.

poling with green wood. I have also experienced difficulty in obtaining samples of overpoled copper free from sulfur in small test bars, though standard 200-lb. wire-bars cast from the same melt spewed badly. I would, therefore, venture to suggest that had Mr. Skowronski been able to make a larger casting, or to eliminate the quick chill of his small test-bar mold, he would have found indications of overpoling in duplicate tests even though sulfur was not present.

Some years ago, I had occasion to make a test along somewhat similar lines. Owing to the difficulty, at that time, of obtaining accurate laboratory results on minute quantities of sulfur, in the neighborhood of our large refineries, sulfur analyses were omitted. A 100,000-lb. furnace was used and a semi-bituminous coal of exceptionally low sulfur content was selected for fuel. The cathodes charged were carefully washed twice with a finely divided high-pressure stream of warm water to remove the last traces of copper sulfate solution. The cathodes were charged and melted as rapidly as possible, care being taken to maintain an oxidizing atmosphere in the forehearth of the furnace while melting. The copper was oxidized to "set copper" and poling was begun. While poling, the metal bath was kept completely covered with clean lump charcoal. Samples were taken at approximately 10-min. intervals and analyzed for copper, the first sample being taken just before level set was reached. Poling was continued after "tough pitch" was reached and samples of the overpoled copper were carefully analyzed. Care was taken during the poling period to avoid adding fresh fuel to the fire. Results were as follows:

Sample No.	Per Cent Cu		Sample No.	Per Cent. Cu.	
1	99.860	low set	6	99.959	high set
2	99.890	low set	7	99.978	high ridge
3	99.934	level set	8	99.957	spewed badly
4	99.941	level set	9	99.934	spewed badly
5	99.953	slightly convex			

Samples for analysis were small flat dip samples designed to avoid segregation. Castings made for the observation of "set" were standard 135-lb. wire-bar.

Owing to the precautions taken to exclude sulfur as a factor, *i.e.*, extra washing of the cathodes, thorough oxidation of the charge, the use of charcoal as a covering for the bath, the avoidance of fresh fuel during the poling period, and the low sulfur content of the coal, the decrease in the percentage of Cu in the castings was attributed to the absorption of reducing gases from the green wood, probably CO and H, which, on segregating in the castings, were not entirely given off. The same result

could, of course, have been due to the absorption of  $\text{SO}_2$ , but in this case, to have attributed the falling off in the percentage of Cu to that cause would have meant the absorption of at least 22 lb. of sulfur in approximately 20 minutes.

In conclusion, I would state that I have never found it impossible to overpole electrolytic copper under manufacturing conditions; also, that the amount of oxygen remaining in refined copper, to neutralize the effects of sulfur and other reducing gases present, will vary considerably with the amount of segregation which takes place in the casting. It also follows that castings designed to eliminate segregation will, in commercial practice, contain a higher percentage of Cu than those in which, on account of their shape, segregation is more marked.

F. JOHNSON,\* Birmingham, England (written discussion†).—Mr. Skowronski's first melting experiments tend to show that ingots with a "level set" may be obtained without oxygen. Now it is characteristic of all metals which occupy a smaller volume when solid than they do when molten to "pipe." In vertical-cast ingots, this pipe resembles a horn-shaped cavity, with the wide mouth uppermost. In horizontal-cast ingots, the pipe is a longitudinal furrow along the centerline of the ingot surface.

If copper be successfully deoxidized *and degasified*, this furrow will inevitably be produced. In the author's oxygen-free ingots, it is clear that gases were occluded and these gases effectively opposed the formation of a pipe or furrow by shrinkage (fluid contraction). It is not a special property of any particular gas which produces this effect. Hence it is not surprising that the author has succeeded in producing level and overpoled ingots through the agency of sulfur dioxide gas. That he has counteracted the overpoling influence of sulfur by means of oxygen is somewhat difficult to explain, since the well known reaction between cuprous sulfide and cuprous oxide should result in the production of sulfur dioxide gas. Why the presence of excess cuprous oxide should neutralize the overpoling influence of this gas without eliminating sulfur is not at all clear. It is to be regretted that the oxygen was not determined in the bars given in Tables 3 and 4.

It is by no means established that sulfur is responsible for the overpoling of ingots given in Table 4, since carbon was used for overpoling. If, however, the same kind of poling treatment was given as that applied in the case of ingots in Table 1, the responsibility of sulfur assumes more definite shape.

The author rightly points out that Mr. Johnson's theory of the equilibrium between gases and cuprous oxide does not explain why a

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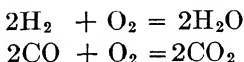
\* Head of Metallurgy Department, Municipal Technical School.

† Received Sept. 12, 1918.

simple addition of cuprous oxide does not correct an overpoled charge. Such a charge must be re-worked to "set" copper. This re-working (rabbling) undoubtedly forcibly removes the excess gases, which are not removable by cuprous oxide.

The writer suggests that the gases concerned in the production of "tough-pitch" copper are of two kinds, soluble and insoluble. The soluble gases, such as hydrogen and possibly carbon monoxide, may be capable of being retained in solid solution and therefore would, if wholly in this condition, play no part in the formation of "set."

In the absence of other gases, and of cuprous oxide, such copper should set with a depression and give characteristically underpoled ingots. The introduction of cuprous oxide is followed by a reaction, for example:



Thus insoluble gases are formed which cannot be retained in solid solution and which, in endeavoring to escape before solidification is complete, will oppose themselves to the natural shrinkage of the metal and result in the production of ingots with level surfaces (tough-pitch) or of overpoled ingots, if in excess. The action of overpoling will thus be the production of more soluble gases which react with cuprous oxide and produce an accumulation or excess of insoluble gases. This excess would cause spewing.

The writer has no experimental evidence to offer in support of this theory, but the inability of cuprous oxide to neutralize the overpoling action of insoluble gases is thus explained. Their removal must be effected mechanically.

The writer has found, in his experience, that it is possible to produce ingots of electrolytic copper which are so overpoled as to spew even on an experimental scale. The copper used was cathode copper of high electrical conductivity (over 101 per cent.); sulfur was not determined.

S. M. Hopkins, metallurgist at the Birmingham Battery Co., and a copper refiner of many years' experience, confirms this.

## Action of Reducing Gases on Hot Solid Copper

BY NORMAN B. PILLING,\* M. S., PITTSBURGH, PA.

(Milwaukee Meeting, October, 1918)

The deleterious effect on the mechanical properties of copper, resulting from heating in contact with reducing gases, is well known, but the mechanism of the action does not appear to have been definitely established. The purpose of the present investigation was not to determine the exact variations in physical quality of commercial copper, with its various impurities, as dependent upon the composition of the gas, temperature of exposure, etc., but rather to study the nature of the action and the conditions under which it occurs.

The physical result of the so-called "gassing" of copper has been generally described as the development of "brittleness." There appears to be a striking decrease in tensile strength, associated with a considerable loss of ductility, and a small copper strip or wire affected in this way can be snapped in two when bent. Larger articles may have an outer layer alone affected, which can be detached from the core by hammering or bending.

Perhaps the earliest reference to this subject is that of Heyn<sup>1</sup> in 1900, and among the later observers may be mentioned Archbutt,<sup>2</sup> Bengough and Hill,<sup>3</sup> Johnson,<sup>4</sup> Mathewson and Thalheimer,<sup>5</sup> and Ruder.<sup>6</sup> Bengough and Hill, while working with arsenical copper, were the first to suggest the possibility of the formation of gases within the copper itself, while Ruder endeavored to explain the action by the reduction of the

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\* Metallurgist, research laboratory, Westinghouse Elec. & Mfg. Co.

<sup>1</sup> E. Heyn: Die Umwandlung des Kleingefüges bei Eisen und Kupfer durch Formänderung im kalten Zustande und darauf folgendes Ausgluhen. *Zeitschrift vereines Deutscher Ingenieure* (1900) **44**, 508.

<sup>2</sup> L. Archbutt: Determination of Oxygen in Copper. *Analyst* (December, 1905) **30**, 385..

<sup>3</sup> G. D. Bengough and B. P. Hill: Properties and Constitution of Copper-arsenic Alloys. *Journal, Institute of Metals* (1910) **3**, 34.

<sup>4</sup> F. Johnson: A Method of Improving the Quality of Arsenical Copper. *Journal, Institute of Metals* (1913) **10**, 275.

<sup>5</sup> C. H. Mathewson and E. M. Thalheimer: Comparisons between Electrolytic Copper and Two Varieties of Arsenical Lake Copper with Respect to Strength and Ductility in Cold-worked and Annealed Test Strips. *Trans.* (1916) **55**, 446.

<sup>6</sup> W. E. Ruder: The Brittleness of Annealed Copper. *Trans. American Electrochemical Society* (1916) **29**, 515.

cuprous oxide, leaving a spongy mass having little mechanical strength. The possibility of a direct action of the hydrogen on the copper, forming hydrides, has received but scant support. Quantitative measurements of the effect of the action on the strength and other properties of electrolytic and arsenical copper may be found in the papers by Bengough and Hill, and Mathewson and Thalheimer, cited above.

For the sake of simplicity, attention has here been confined mainly to commercial-conductivity copper, non-arsenical, with 0.05 per cent. oxygen, and about 0.015 per cent. lead. A preliminary study of the effects of reducing gases on hot copper was made by passing the desired gas over a length of soft copper strip heated in an electric furnace. After 30-min. exposure to hydrogen at 800° C. there was a marked deterioration in the quality of the copper. Whereas initially it was quite ductile and resistant to bending, after this treatment it was extremely weak and friable, as evidenced by its inability to withstand even slight bending.

After similar treatment in natural gas and in carbon monoxide, the same phenomenon was observed, but the deterioration was less strongly marked with the latter gas. Similar treatment in an atmosphere of steam caused no observable change in physical quality.

The microscopic appearance of the copper before treatment is shown in Fig. 11; it is homogeneous, with a uniform distribution of  $\text{Cu}_2\text{O}$  particles. After heating in hydrogen, it has become very porous (Fig. 12), each  $\text{Cu}_2\text{O}$  particle has been replaced by a void and, in addition, there are large angular cracks which, as shown by etching, follow the grain boundaries. This observation agrees with the micrographs given by previous investigators. The copper samples heated in natural gas and in carbon monoxide were similar in appearance, but the action was less marked. In no cases could unreduced grains of  $\text{Cu}_2\text{O}$  be detected.

#### PHYSICAL EFFECT OF HYDROGEN ON COPPER

From the results of preliminary experiments, hydrogen seemed to be the most active of the several reducing gases tried, and it appeared likely that in a mixture of such gases hydrogen would play the predominant part. It is likely that any hydrocarbons present would decompose at the temperatures used, and the action would be traceable to the simple gases formed ( $\text{H}$ ,  $\text{CO}$ , etc.).

The physical effect of heating copper in contact with hydrogen was studied with a number of copper strips,  $\frac{5}{8}$  by 0.048 in. (15.9 by 1.2 mm.). The oxygen content of this copper was 0.05 per cent. and the material, received in a dead soft condition, showed satisfactory physical properties. These strips were electrically heated at various temperatures in a small tube furnace, evacuated to prevent excessive oxidation, and after the desired temperature had been attained, as measured by a small Pt-



PtRh thermocouple lying at the center of the piece, hydrogen was admitted and passed through the furnace tube over the strip for 30 min. At the end of that time the strip was withdrawn and cooled in the air. A rough measure of the effect of this treatment was made by noting the number of  $180^\circ$  bends over a radius of about  $\frac{1}{4}$  in. (6.3 mm.) the strip would stand before breaking. The effect of complete hydrogenation is so marked that this was found to be a sufficiently sensitive test, though giving comparative values only. The following table gives the results obtained, which are plotted in Fig. 1.

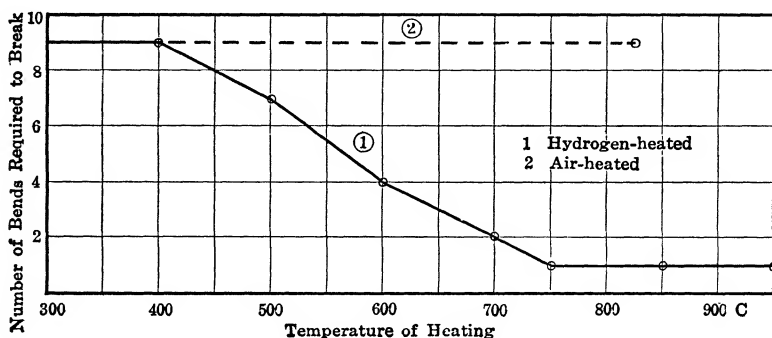


FIG. 1.—EFFECT OF HYDROGEN ON COPPER STRIPS. (Length of heating, 30 min.)

TABLE 1.—*Effect of Heating Copper Strips in Hydrogen*

Temperature	No. Bends to Break	Temperature	No. Bends to Break
(As received)	9	750°	1
400°	9	850°	1
500	7	950	1
600	4	1050	1
700	2		

That this deterioration in quality was due to the hydrogen and was not an annealing effect was shown by heating similar strips at  $400^\circ$  and  $800^\circ$  in air (reduced pressure), after which no change in tenacity could be observed.

It will be noticed that the deterioration starts between  $400^\circ$  and  $500^\circ$  C.—well below a red heat—and increases in severity as the temperature of heating becomes higher. An idea of the rate at which this action takes place may be obtained from Fig. 2, in which the bending qualities, after exposure to hydrogen for varying lengths of time at  $800^\circ$  are plotted. In 10 min. the deterioration is complete.

Typical examples of the structures encountered after hydrogenation are shown in Figs. 12 and 13.

#### NATURE OF HYDROGENATION

The mechanism of the action during exposure of copper to hydrogen appears to be as follows: Commercial-conductivity copper contains various impurities, all, except oxygen and occasionally lead, being measured in thousandths of a per cent. Oxygen normally occurs from 0.03 to 0.08 per cent. in the form of  $\text{Cu}_2\text{O}$  mechanically mixed with the metallic copper. The presence of this quantity of  $\text{Cu}_2\text{O}$  seems to be necessary, in order to avoid the reduction of metallic impurities in the slag from the oxidized to the metallic condition, during refining, and to promote soundness in casting. If hydrogen is physically soluble in solid copper at high temperatures, this gas would penetrate the metal, attack and re-

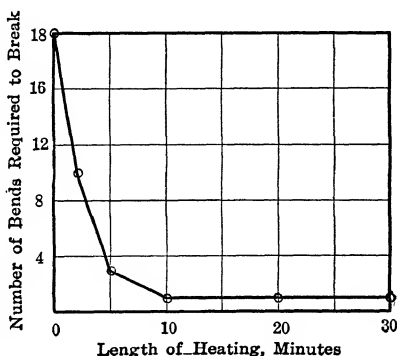
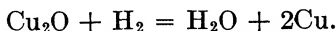


FIG. 2.—EFFECT OF HYDROGEN ON COPPER STRIPS AT 800° C.

duce the grains of  $\text{Cu}_2\text{O}$ , with the formation of steam, according to the equation



After this reduction, the volume of the copper is but 60 per cent. of the volume of the  $\text{Cu}_2\text{O}$  from which it was reduced, thus leaving voids marking the site of the original oxide granules and approximating 40 per cent. of the space formerly occupied by them.

If steam is physically less soluble in copper than hydrogen, it will accumulate in these pockets at a rate faster than it can dissolve in the copper and diffuse away, and the net result of the reaction would be the formation of a quantity of steam within the voids left by the  $\text{Cu}_2\text{O}$ , and at considerable pressure. A rough calculation of the magnitude of the pressure possible at 800° C., assuming the steam to be completely insoluble in the copper, showed that 8000 to 9000 atm. would develop

if there were no yielding in the copper. The investigations of Bengough<sup>7</sup> on the tensile properties of copper at high temperatures have shown that as the temperature rises the tensile strength falls off rapidly until a "mechanical critical temperature" is reached between 600° and 750° C., varying apparently with the nature and quantity of impurities present. Above this temperature the tensile strength diminishes linearly to zero at the melting point, and in this range the copper is characterized by extreme intercrystalline weakness. Now the combination of considerable internal pressure (steam), with lack of intercrystalline cohesion, forces the grains apart until relief is obtained (see Fig. 18). Deoxidation alone cannot account for the weakness developed, for it is not clear why the substitution of a small void for a small particle of very brittle cuprous oxide should weaken the surrounding metal. A careful examination of copper strips heated at 800° in hydrogen showed that a mechanical swelling takes place coincident with the formation of intercrystalline ruptures. The strip increased in thickness by 4 per cent. and in width by 1.5 per cent. while decreasing about 4 per cent. in density. This decrease in density is not due solely to voids left in the metal by the reduction of the cuprous oxide, as the decrease noted was 130 times as great as the observed loss in weight due to removal of oxygen. The resultant weakness of the copper after this action is due simply to the absence of mechanical coherence between individual grains within the puffed copper.

#### PERMEABILITY OF COPPER TO HYDROGEN, STEAM, CARBON MONOXIDE AND CARBON DIOXIDE

In order to verify the explanation given for the "puffing" of copper, the relative permeabilities of several reducing gases and their oxides were determined. The method employed was to observe the change in pressure within an evacuated thin-walled copper tube heated in an atmosphere of the gas in question, in a nichrome-wound quartz-tube furnace. The copper tube extended throughout the length of the furnace, one end of it being pinched and sealed with solder, and the other waxed to the glass vacuum system. An optical-lever manometer<sup>8</sup> was used to measure the slight increments in pressure over the initial pressure (5 mm.), and by reducing the volume auxiliary to the copper tube to the least possible amount, a sensitive arrangement was obtained. In

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<sup>7</sup> G. D. Bengough: A Study of the Properties of Alloys at High Temperatures. *Journal, Institute of Metals* (1912) 7, 123.

G. D. Bengough and D. Hanson: Tensile Properties of Copper at High Temperatures. *Journal, Institute of Metals* (1914) 12, 56.

<sup>8</sup> A modification of that described by J. E. Shrader at the Pittsburgh meeting of the American Physical Society, Dec., 1918.

use, both the copper tube and the enclosing furnace tube were evacuated until a steady temperature was reached, measured with a small Pt-PtRh couple adjacent to the copper tube, and then the desired gas was introduced, flowing slowly through the quartz furnace tube at atmospheric pressure. This arrangement is shown in Fig. 3.

The hydrogen and carbon dioxide were taken directly from pressure tanks; the carbon monoxide was generated by the action of sulfuric and formic acids, the gas being dried before entering the furnace; the steam was introduced wet at atmospheric pressure.

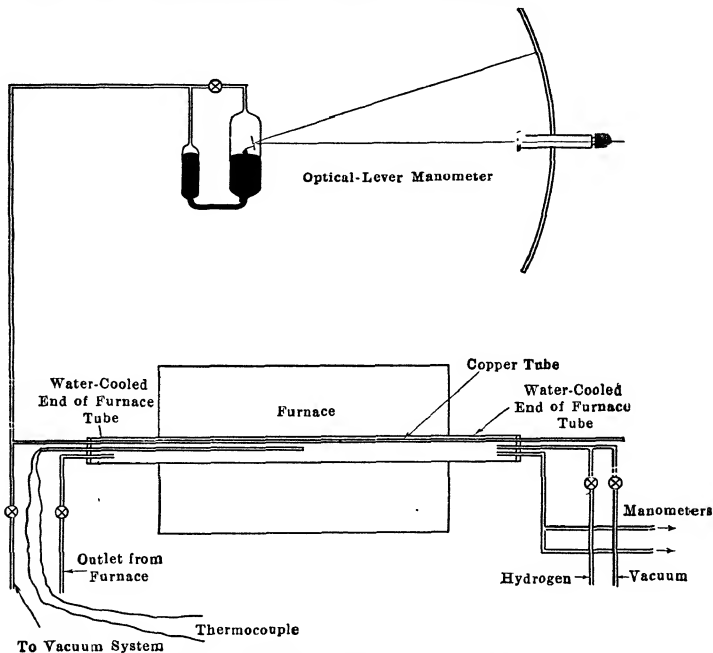


FIG. 3.—ARRANGEMENT OF APPARATUS.

After some experimentation, 700° C. was chosen as a temperature suitable for making measurements of the comparative rates of diffusion of these gases through copper. Curves giving the observed change in pressure inside the copper tube, due to diffusion of the gases through its walls, are shown in Fig. 4. The following table gives comparative measurements, the rate of diffusion being taken as the maximum slope of the pressure-time curve, referred to hydrogen as 1000 units.

TABLE 2.—Rate of Diffusion of Gases Through Copper at 700° C.

GAS	DIFFUSION RATE
H <sub>2</sub> .....	1000
H <sub>2</sub> O.....	65
CO.....	17
CO <sub>2</sub> .....	0.6

It will be seen that hydrogen will diffuse into copper approximately 15 times as fast as water, formed by the reaction of hydrogen on  $\text{Cu}_2\text{O}$ , can diffuse out; and similarly, that carbon monoxide diffuses through copper about 25 times faster than the carbon dioxide formed when it reduces the cuprous oxide.

This result seems to confirm the above explanation, that the weakness experienced by copper containing disseminated oxide, after exposure to reducing gases at high temperatures, is due to the internal fractures pro-

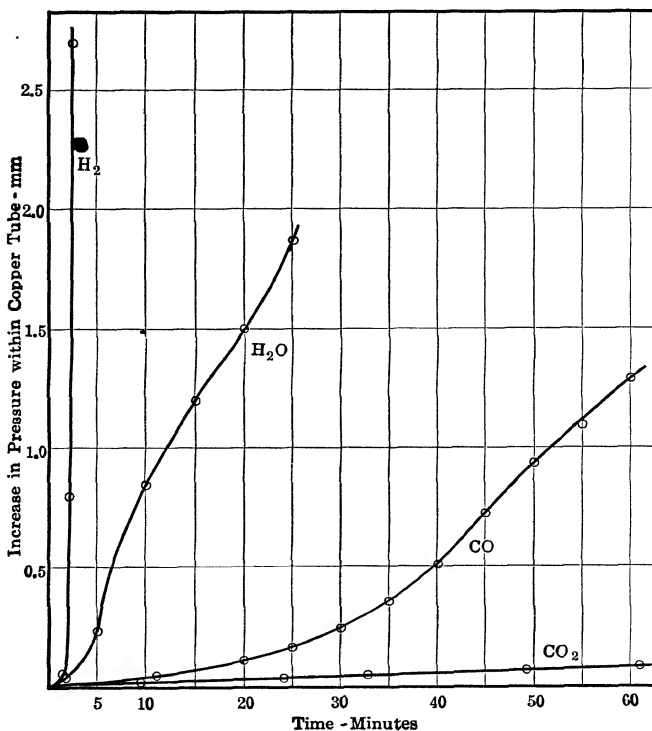


FIG. 4.—DIFFUSION OF HYDROGEN, STEAM, CARBON MONOXIDE, AND CARBON DIOXIDE THROUGH COPPER AT  $700^\circ\text{C}$ .

duced by the expansive action of a gas which is formed by the reduction of each oxide granule at a rate much faster than that at which it can dissolve in the copper and diffuse away.

An interesting check on the regularity of the measurements is that if the logarithm of the rate of diffusion, as found above, is plotted against the molecular weight of the gas, a straight line is obtained.

The transmission of hydrogen through copper was further studied by making diffusion measurements at a number of temperatures. The arrangement of the apparatus was the same as that described above, except that at temperatures above  $700^\circ$ , when diffusion was rapid

and the pressure change considerable, a direct-reading mercury manometer was used. The results, shown graphically in Figs. 5 and 6, are as follows, the rate at 700° being considered unity.

TABLE 3.—*Diffusion of Hydrogen Through Copper*

TEMPERATURE	RATE OF DIFFUSION
950° C.....	17.0
850.....	10 0
750.....	2.6
700.....	1 0
500.....	0 54
400.....	0 006

Plotting the rate of diffusion against temperature, the curve (Fig. 7) first shows appreciable diffusion between 400° and 500°, and rises

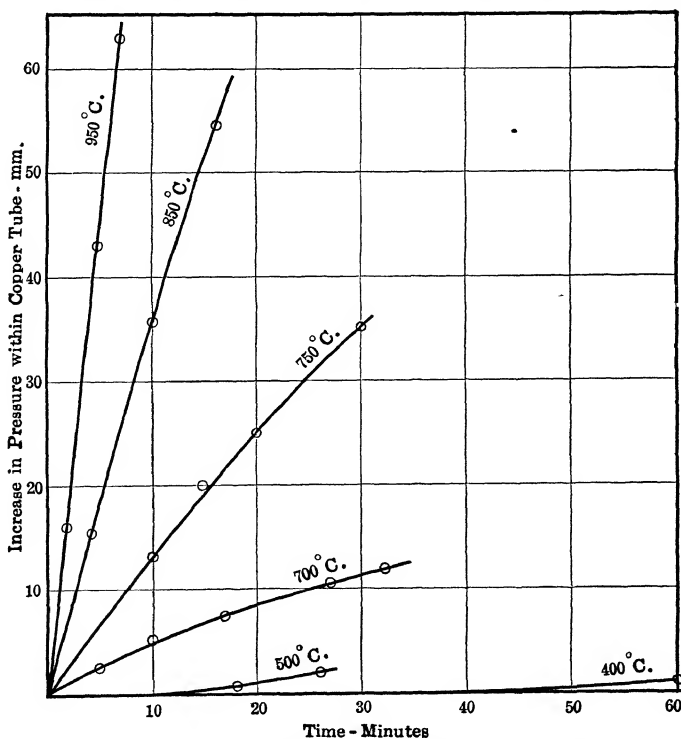


FIG. 5.—DIFFUSION OF HYDROGEN THROUGH COPPER.

sharply beyond 700°. This agrees with the observations made on the physical effects of hydrogenation, recorded above, in which incipient deterioration in quality was noted in this same temperature interval. The diffusion rate measured at 800° and above is probably in excess of the

true rate of diffusion, due to the known fact that at these temperatures the fissures produced by the reaction on the oxide are of sufficient size to make the tube permanently porous.

### REDUCTION OF CUPROUS OXIDE BY HYDROGEN

To check the dependence of hydrogenation upon the diffusion rate of hydrogen through copper, a series of solid copper strips were exposed to hydrogen at temperatures between 200° and 900° for varying lengths of

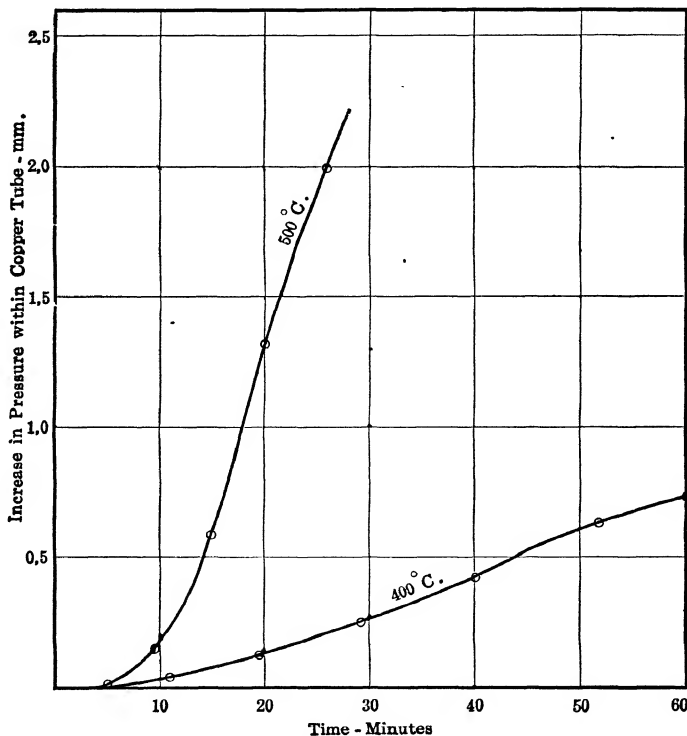


FIG. 6.—DIFFUSION OF HYDROGEN THROUGH COPPER.

time. Since cuprous oxide is readily reduced to metal by hydrogen at 147° C.,<sup>9</sup> it is evident that the depth to which the reduction of the oxide granules has taken place is a measure of the depth to which the hydrogen has diffused, assuming that the hydrogen concentration necessary before reduction takes place is small. Cross-sections of these copper pieces were made and the depth of deoxidation was measured. It was found that the deoxidized envelope surrounding the central oxide-containing core, while not sharply defined, was of rather uniform thickness in indi-

<sup>9</sup> C. N. Otin: *Metallurgie* (1912), 9, 98.

vidual samples. A representative example of this penetration is shown in the micrograph, Fig. 14. The curves in Fig. 8 show how the depth of this layer varies with the time of exposure. There was no observable

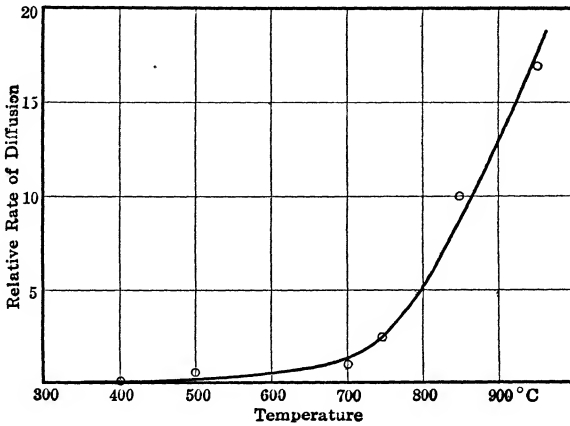


FIG. 7.—RATE OF DIFFUSION OF HYDROGEN THROUGH COPPER.

reduction of internal oxide particles at 400° after an hour's exposure, nor at any temperature below this. Taking the values of the initial slopes of this family of curves as proportional to the rate of diffusion,

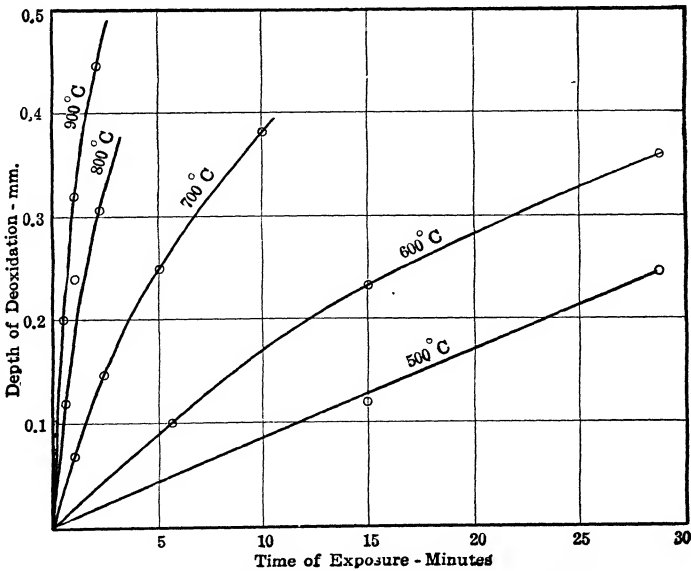


FIG. 8.—PENETRATION OF HYDROGEN INTO COPPER.

and plotting these values against the temperature, a curve is obtained (Fig. 9) very similar to the diffusion curve as measured by the copper tubes, but not influenced by the opening of minute crevices as the reduc-



tion of oxide proceeds. Active diffusion becomes apparent between 400° and 500° C.

A comparison between the two determinations is given in Fig. 10,

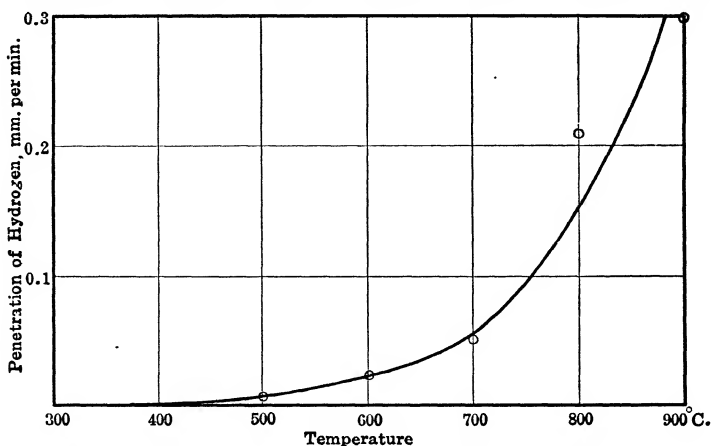


FIG. 9.—RATE OF DIFFUSION OF HYDROGEN INTO COPPER.  
(Using reduction of cuprous oxide particles as indicator.)

where the two diffusion curves are plotted to the same scale, *i.e.*, relative rates of diffusion compared with the rate at 700° as unity. The divergence above 700°, due to the influence of the increasing porosity of the copper tubes, is apparent.

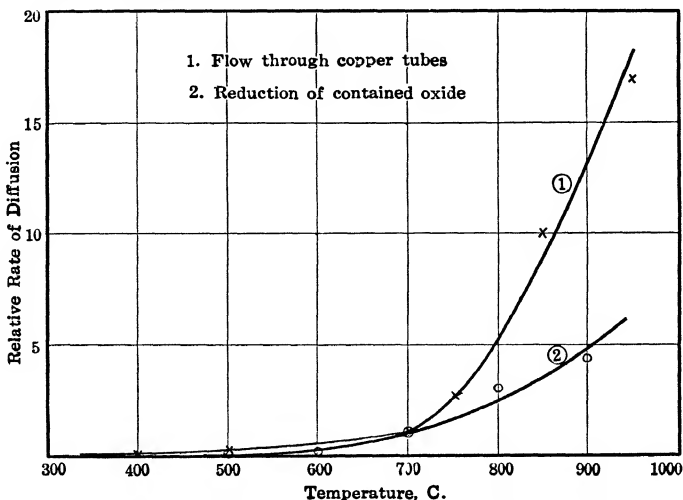


FIG. 10.—RELATIVE RATES OF DIFFUSION OF HYDROGEN THROUGH COPPER.

#### INFLUENCE OF OXYGEN CONTENT

The oxygen content of the copper strips used in all the foregoing work was 0.05 per cent. Copper strips containing larger quantities of

oxygen were obtained, and were given similar exposures to hydrogen, *i.e.*, 30 min. at 800°. A quantitative measure for the disruption of the copper is not easy to find, but an examination of the micrographs, Figs. 15 to 18, shows that as the oxygen content increases, the severity of the action also increases. The photographs are from unetched surfaces and the grain outlines are due wholly to the intercrystalline ruptures. A consideration of what happens during hydrogenation shows that it is not oxygen content alone that determines the severity of the action. The reaction that takes place when a particle of cuprous oxide is reduced to metallic copper is quantitative; the volume of the resulting void is definite, depending on the specific volumes of copper and cuprous oxide, and the quantity of water formed, existing as a gas and filling this space, is also definite. At a given temperature the ratio of these two quantities determines the pressure, but this ratio is independent of the total quantity of oxide present. The actual pressure, then, is a function of the temperature.

At high temperatures the cohesion\* between two copper grains is much less than the point to point cohesion within one of them, so that it is only the gas-filled pockets that happen to lie along a grain boundary that are operative in causing rupture. Since this is so, cast copper, in which all the oxygen present is concentrated along the grain boundaries as a cuprous oxide-copper eutectic, is peculiarly susceptible to hydrogenation. This is strikingly shown in Figs. 19 and 20, which is a cast copper containing but 0.02 per cent. oxygen, and Fig. 21, showing a piece of cast copper containing 0.50 per cent. oxygen (more than the eutectic concentration) before and after hydrogenation. In the latter case, the explosive action of the gas formed has completely shattered the metal.

In wrought copper, during the process of rolling, the oxide particles become thoroughly mixed with the metal, the recrystallization taking place with complete disregard of their presence, so that the situation of an oxide granule at a grain boundary is governed simply by chance. An increased oxygen content increases the severity of hydrogenation in wrought copper only because of the greater possibility of oxide granules to lie along the grain boundaries.

From the nature of the action which causes the mechanical deterioration, it is not expected that any chemical or heat treatment short of remelting would restore hydrogenated copper to its former condition.

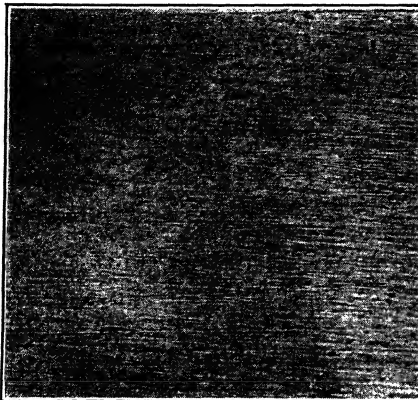


FIG. 11.—ROLLED COPPER,  $O = 0.05$  PER CENT. UNTREATED,  $\times 100$ .



FIG. 12.—SAME AS FIG. 11, HEATED 30 MIN. AT  $800^{\circ}\text{C}$ . IN HYDROGEN.  $\times 100$ .

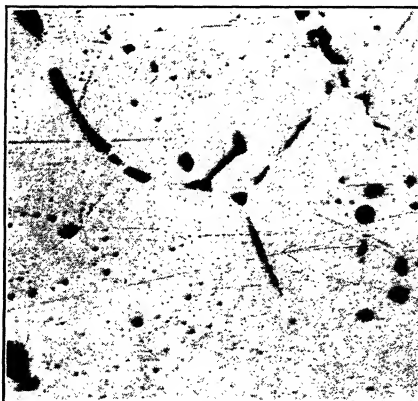


FIG. 13.—SAME AS FIG. 11, HEATED 30 MIN. AT  $1050^{\circ}\text{C}$ . IN HYDROGEN.  $\times 100$ .

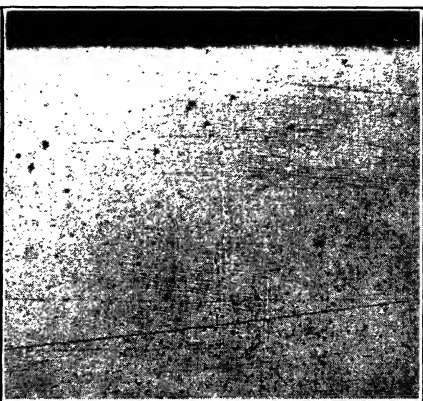


FIG. 14.—PENETRATION OF HYDROGEN INTO COPPER. EXPOSED TO HYDROGEN 2 MIN. AT  $800^{\circ}\text{C}$ .  $\times 100$ .

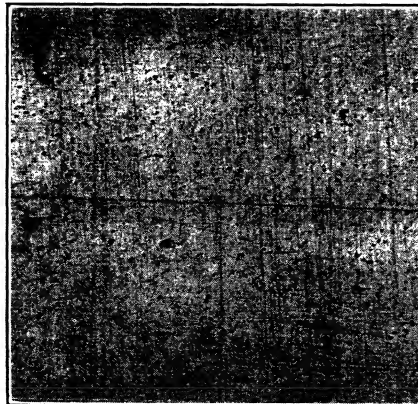


FIG. 15.—ROLLED COPPER,  $O = 0.09$  PER CENT. UNTREATED.  $\times 100$ .

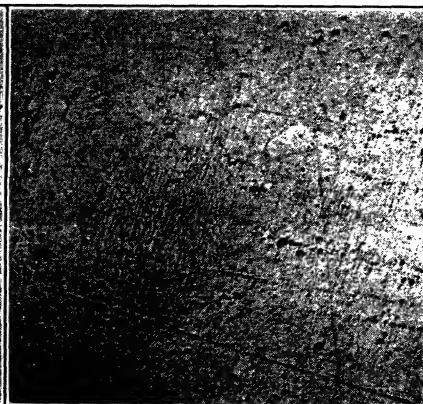


FIG. 16.—SAME, HEATED 30 MIN. AT  $800^{\circ}\text{C}$ . IN HYDROGEN.  $\times 100$ .

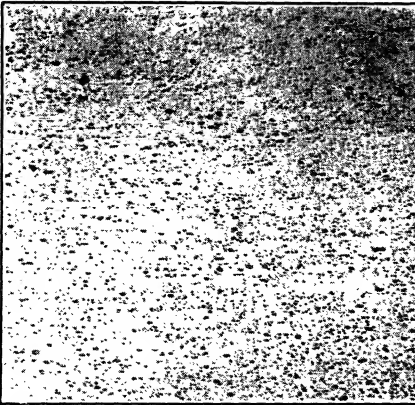


FIG. 17.—ROLLED COPPER,  $O = 0.21$  PER CENT. UNTREATED.  $\times 100$ .

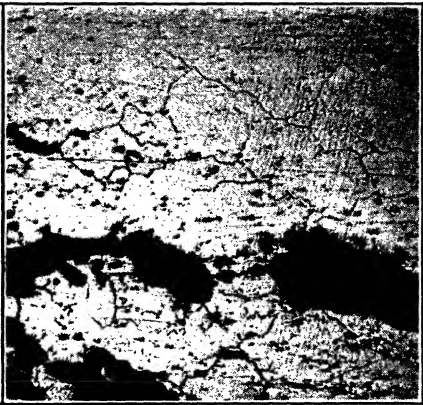


FIG. 18.—SAME, HEATED 30 MIN. AT  $800^{\circ}\text{C}$ . IN HYDROGEN.  $\times 100$ .

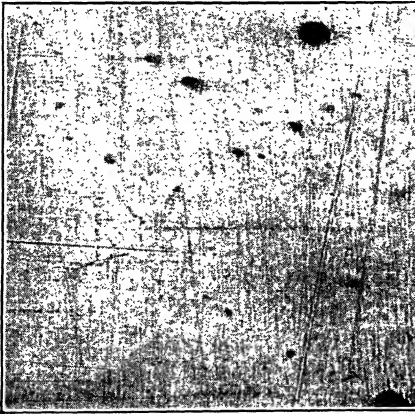


FIG. 19.—CAST COPPER,  $O = 0.02$  PER CENT. UNTREATED.  $\times 100$ .

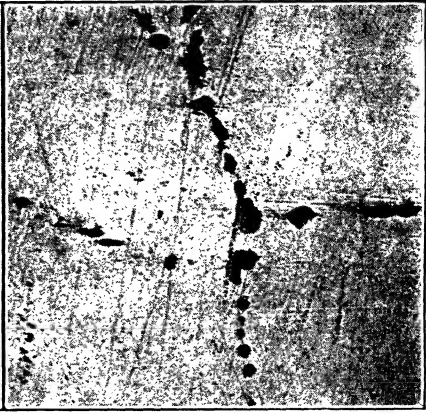


FIG. 20.—SAME, HEATED 30 MIN. AT  $800^{\circ}\text{C}$ . IN HYDROGEN.  $\times 100$ .

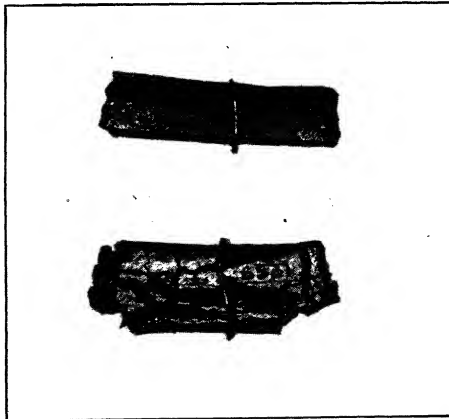


FIG. 21.—CAST COPPER,  $O = 0.50$  PER CENT., BEFORE AND AFTER HEATING IN HYDROGEN AT  $850^{\circ}\text{C}$ . NATURAL SIZE.

## DISCUSSION

W. H. BASSETT,\* Waterbury, Conn. (written discussion †).—The effect of reducing gases on hot solid copper has been known for many years in the copper industry, and precautions taken to guard against it in the general practice in copper heating and annealing. In view of the statement that Bengough and Hill, in 1910, were the first to suggest the possibility of the formation of gas within the copper itself, it is interesting to note that in *Harper's Magazine* for April, 1904, an article, "Life and Diseases of Metals," by Professor Heyn, illustrated the effect of reducing gases on copper by a photograph, which is shown in Fig. 1. This he entitled "Copper burst asunder by disease."

After referring to the effect of hydrogen on steel, he makes the following remark:

"Similar symptoms of poisoning, caused by hydrogen or gases containing hydrogen (as gas for lighting purposes), are apparent in copper when exposed to red heat. Not every kind of copper is susceptible to this poisoning in equal degree. Copper perfectly free of cuprous oxide is entirely exempt from poisoning. Most of the various coppers of commerce, however, contain cuprous oxide, formed during the smelting process while exposed to atmospheric influences. In such coppers, containing cuprous oxide, hydrogen causes a terrible disease on the copper being heated red hot. The copper bursts asunder and is permeated by cracks. This disease is practically incurable and can be eradicated by resmelting only. The results work destructively according to the amount of cuprous oxide contained in the copper."

It must have been evident to Professor Heyn, as it was to us on reading this article, that the bursting asunder of the copper was due to the formation of steam. Practical copper heaters have known for many years that it was quite possible to produce blisters, or large gas holes, in the interior of copper plates or bars by heating to a high temperature in reducing gases. These facts, of course, do not detract from the value of the study of the action of hydrogen gas that has been made by Mr. Pilling, but simply illustrate that the practical effect of reducing gases on furnace-refined copper has long been a troublesome factor in the heating or annealing of that metal.

It would be very interesting to know what Mr. Pilling means by "commercial-conductivity" copper, since the usual terms under which copper is specified are electrolytic or lake copper; the lake being divided into two grades known as low-resistance, or high-conductivity, and high-resistance, or low-conductivity copper. The electrolytic and low-resistance lake copper are both non-arsenical.

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\* Technical Superintendent and Metallurgist, The American Brass Co.

† Received Nov. 27, 1918.

The lead figure given is somewhat surprising, because copper with 0.15 per cent. lead could be hot rolled only with great difficulty. This amount of lead would result in the serious cracking of edges in plates and cross cracking in rods. Ordinarily good electrolytic or lake copper does not carry more than 0.001 or 0.002 per cent. of lead.

We cannot quite agree with the author that oxygen normally occurs mechanically mixed with metallic copper in the form of  $\text{Cu}_2\text{O}$ . Furnace-refined copper, whether cast or wrought, is usually thought of as an alloy of copper with cuprous oxide, and the eutectic of copper with cuprous oxide is usually found between the crystals of pure copper, whether in the cast or the wrought form. It is true that there may be inclosures of eutectic within the grains of pure copper, particularly in the wrought material, but there is strong evidence for believing it occurs mainly between the grains. This is indicated in Figs. 2 and 3, which show the surface of wrought copper, in which the grains are strongly outlined, or parted, and where the spots resulting from reduction of suboxide in eutectic appear more frequently on the grain boundaries than elsewhere. This condition is also shown in Figs. 4 and 5, which show a cross-section of a rectangular bar of copper, where the grains are separated in the deoxidized part and the deoxidized part is partly separated from the mass of the material, probably because it occupies a larger volume than it originally did. The effects of rolling material of this kind where the grains have been separated by deoxidation are shown in Fig. 6.

The effect of various gases on refined copper is shown in a series of four micrographs of copper that has been heated to  $800^\circ\text{C}$ . The first, Fig. 7, shows the effect of hydrogen on refined copper; the second, Fig. 8, the want of effect of hydrogen on copper that has been deoxidized by boron-suboxide; the third, Fig. 9, the effect of carbon dioxide on refined strip copper; and the fourth, Fig. 10, the effect of nitrogen on similar material. The larger the amount of oxygen, the greater is the effect of reducing gases. For instance, two lots of strip copper that had been taken from bars coming from the same furnace charge, one containing 0.094 per cent. of oxygen and the other 0.015 per cent. of oxygen, were reduced in tensile strength by annealing for 1 hr. in an atmosphere of illuminating gas at  $600^\circ\text{C}$ . the amounts here shown.

Sample	Original Tensile Strength, Lb. per Sq. In.	Tensile Strength After Gassing, Lb. per Sq. In.	Original Elongation in 2 In. Per Cent.	Elongation After Gassing, Per Cent.
L-1.....	34,000	24,350	36.5	13.0
L-11.....	33,500	28,150	38.0	26.3



FIG. 1.

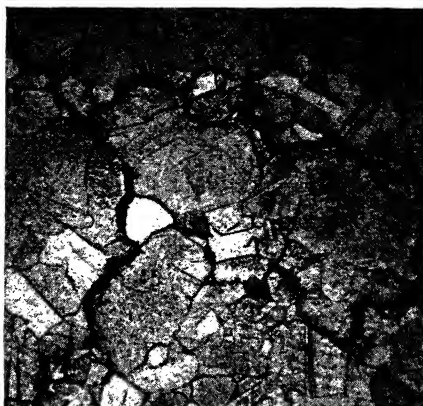


FIG. 2.



FIG. 3.

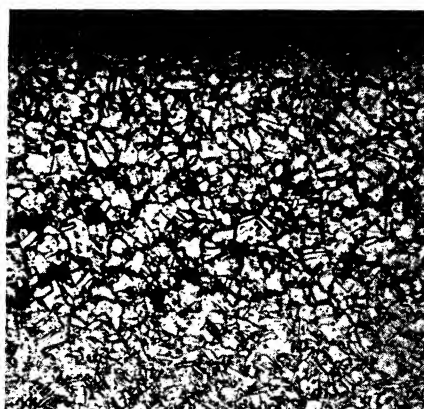


FIG. 4.

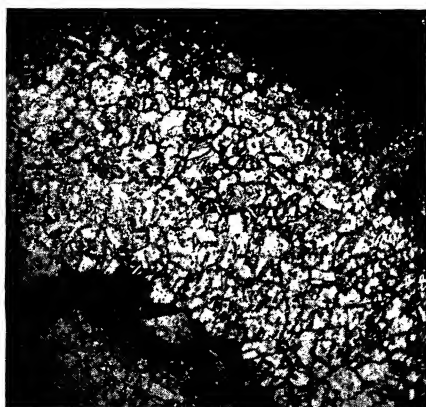


FIG. 5.



FIG. 6.

This indicates a reduction of 28.3 and 16 per cent., respectively, in tensile strength; and 64 and 44 per cent., respectively, in elongation.

The presence of arsenic apparently protects copper from the action of reducing gases to some extent, while the presence of nickel has the



FIG. 7.



FIG. 8.

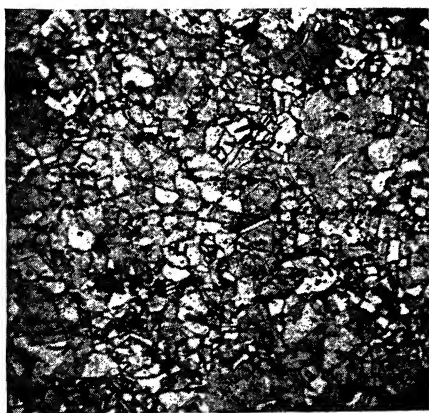


FIG. 9.



FIG. 10.

PLATE II.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

opposite effect. The rate of penetration by reducing gases does not seem to be greatly affected by the amount of oxygen alloy present, as may be noted from Fig. 11, which gives the depth of penetration of both hard and annealed copper rods 0.364 in. in diameter for 1 hr. heating at various temperatures in an atmosphere of illuminating gas.



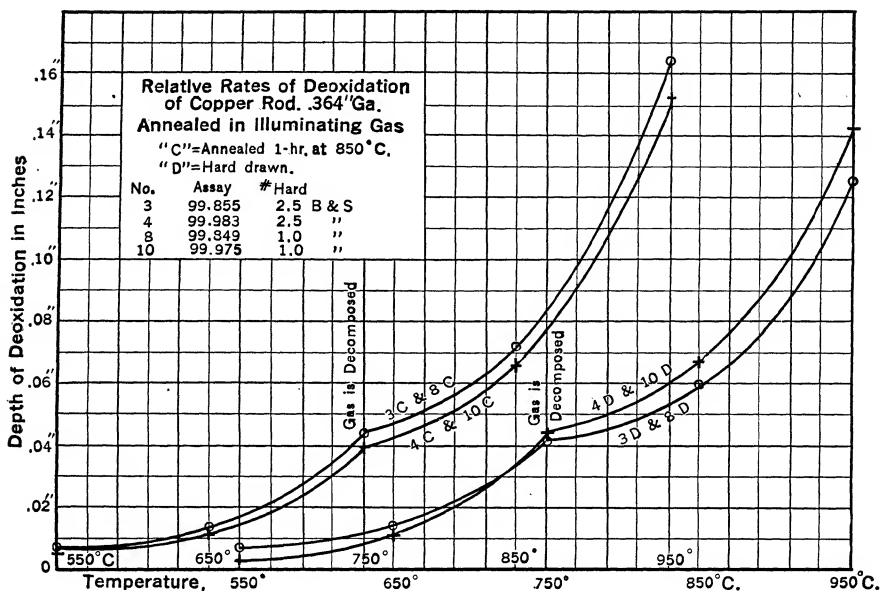


FIG. 11.

NORMAN B. PILLING (author's reply to discussion\*).—The discussion by Mr. Bassett raised one or two points that deserve further discussion. The printed figure for the lead content of the copper used

FIG. 12.—ROLLED AND ANNEALED SHEET. ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

was 0.015 per cent. and not 0.15 per cent., upon which his comment was based. A copper having the latter figure would, of course, be quite unusual. Mr. Bassett takes exception to the writer's statement regarding

\* Received Jan. 16, 1919.

the distribution of cuprous oxide in wrought copper and states that "the eutectic of copper with cuprous oxide is usually found between the crystals of pure copper, whether in the cast or the wrought form." While it is very well known that during the solidification of cast copper the cuprous oxide is segregated completely as an envelope of cuprous oxide-copper eutectic around the crystals of copper free from oxide, it is the writer's opinion that, as a result of repeated rolling, this eutectic structure is quite destroyed and the small particles of cuprous oxide become widely scattered, and in the recrystallization following, during annealing, the rearrangement of the grain boundaries takes place with much indifference to the actual distribution of these particles. In the accompanying micrograph, Fig. 12, taken from a rolled and annealed sheet, the random distribution of the small cuprous-oxide particles with reference to the location of the grain boundaries may be seen.

## Spectroscopic Determination of Lead in Copper

BY C. W. HILL,\* PH. D., AND G. P. LUCKEY, M. A., PITTSBURGH, PA.

(Milwaukee Meeting, October, 1918)

In a previous article<sup>1</sup> preliminary experiments were described, indicating the possibilities of a quantitative spectroscopic method for the determination of small amounts of lead in copper, which would be accurate and rapid, and could be carried out in the refining plant by one not skilled in chemical analysis. The present paper deals with the development of the method in the factory, giving the details of apparatus and its standardization, and presenting a comparison of the accuracy of the method with that of the standard electrolytic determination. (For variations and other applications of the method, the reader is referred to the first article.)

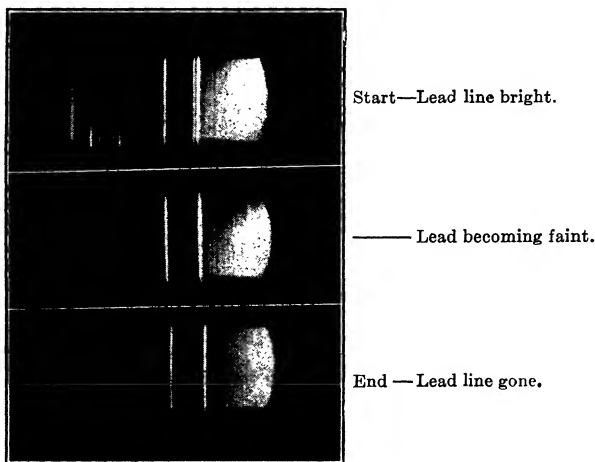


FIG. 1.—STEPS IN THE ELIMINATION OF LEAD.

(The lead line, at point 405.8 in the violet portion of the spectrum, is seen between the two copper lines, 402.29 and 406.29, lying close to the latter. The crowded faint lines are due to the carbon arc.)

### OUTLINE OF METHOD

A carefully weighed sample (0.4 gm.) of the copper to be tested is placed in a cavity in a graphite positive electrode. An arc capable of

\* Research Chemist, Westinghouse Elec. and Mfg. Co.

<sup>1</sup> *Proceedings, American Electrochemical Society* (1918) 32, 335. *Metallurgical and Chemical Engineering* (1917), 17, 659.

regulation is struck, and maintained under constant conditions between the copper sample and a rotating upper carbon electrode. A spectroscope is so adjusted as to observe the lead line ( $405.8\ \mu\mu$ ), as shown in Fig. 1.

With a stop-watch, the time is determined between the melting of the copper sample and the disappearance of the lead line from the spectrum. This time is proportional to the lead content of the sample, the exact values depending upon the apparatus employed. The apparatus

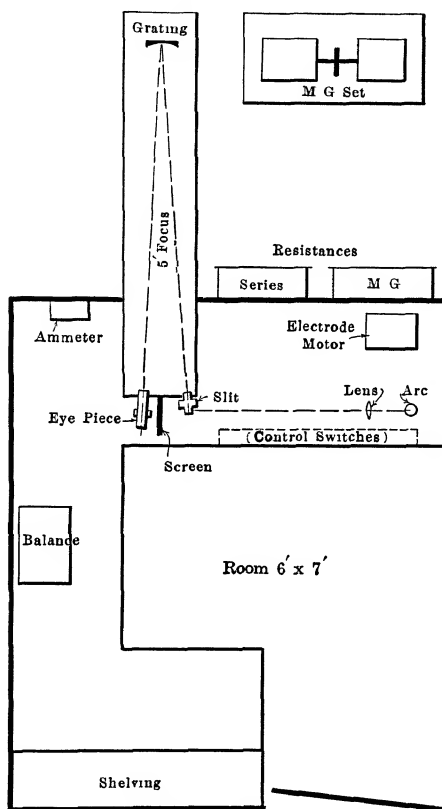


FIG. 2.—PLAN OF TESTING ROOM.

is calibrated with samples of known lead content and a time-lead curve is established for the apparatus. Where minimum values alone are required, the time of disappearance of lead from a sample of satisfactory copper is taken as a standard, and the refining process is continued until furnace samples show a time equal to or less than the standard.

#### APPARATUS

The apparatus and its arrangement as finally installed are shown diagrammatically in Fig. 2 and in the photograph Fig. 3. A full description

of the various parts of the apparatus will be found in Appendix 1. On account of conditions due to the war, we were unable to purchase a spectroscope suitable for the work, and were forced to use a grating, which required more space.

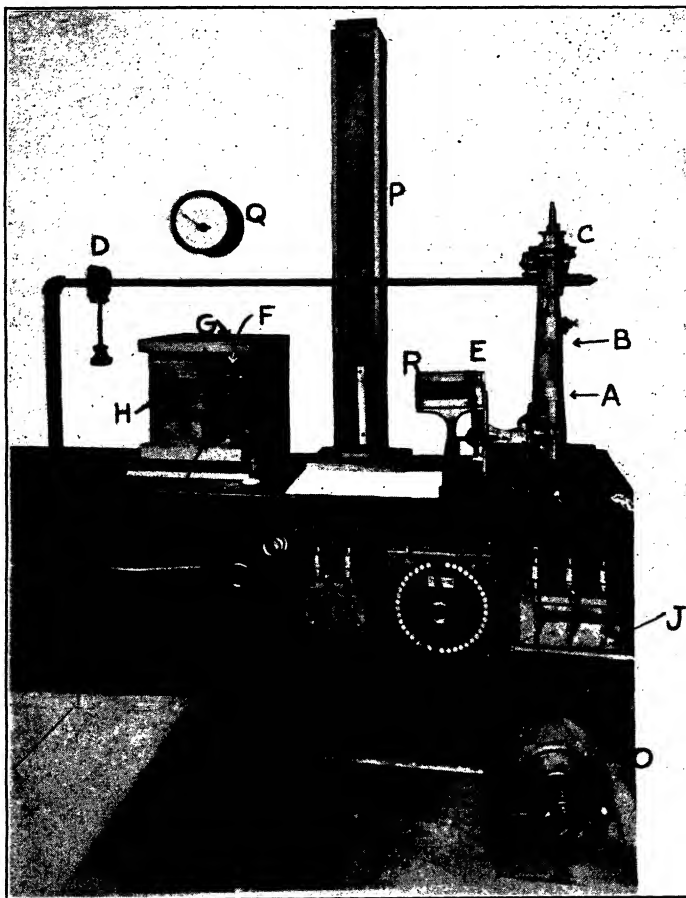


FIG. 3.—ARRANGEMENT OF TESTING APPARATUS.

- |                                |                             |                             |
|--------------------------------|-----------------------------|-----------------------------|
| A—Graphite positive electrode. | G—End of 5-ft. box.         | M—Series resistance knob.   |
| B—Neg. electrode and contact.  | H—Eye-piece.                | N—Rotating electric switch. |
| C—Electrode pulley.            | I—Screen.                   | O—Rotating electric motor.  |
| D—Arc-length control.          | J—Motor-generator switch.   | P—Joly balance.             |
| E—Lens.                        | K—Motor-generator rheostat. | Q—Ammeter.                  |
| F—Prism and slit.              | L—Arc switch.               | R—Colored-glass screen.     |

#### STANDARDIZATION OF APPARATUS

Our first two attempts to standardize the apparatus were not successful. However, we shall give some of the details regarding them, since they throw some light on the relative accuracies of the spectroscopic and electrolytic methods for small quantities of lead. The three methods

of calibration were: (1) checking routine analyses; (2) comparative analyses of large samples; (3) comparative analyses of synthetic samples.

### *Checking Routine Analysis*

For a period of several weeks, the spectroscope was used in comparison with regular routine analyses by the electrolytic method. (Description of method will be found in Appendix 2). The results seemed to check the work of some of the chemists quite accurately, but at best the uncertainty was too great to establish a calibration.

### *Analysis of Large Samples*

Five large samples of copper shot were made by selection from regular furnace runs on the basis of the routine test. These were submitted to three chemists for chemical determination of lead, and were examined in the spectroscope. We were still unable to check the chemical method, and consequently returned the samples for repeat analysis, including repeat samples on regular routine tests. The values obtained are given in Table 1. The analyses on the special samples were made by three chemists, two of them using a 10-gm. sample and one a 25-gm. sample, working independently.

TABLE 1.—*Lead Contents of Special Samples*

No.	Original Anal by 3 Chemists				Check by 3 Chemists				Check by 2 Chemists		
	Av.				Av.				Av.		
1	0.005	0.006	0.005	0.005	.....	.....	.....	.....	0.005	0.005	0.005
2	0.005	0.005	0.007	0.007	0.012	0.012	0.012	0.012			
3	0.010	0.010	0.010	0.010	0.012	0.012	0.015	0.013			
4	0.015	0.015	0.015	0.015	0.013	0.013	0.013	0.013	0.014	0.017	0.015
5	0.020	0.020	0.020	0.020	.....	.....	.....	.....	0.022	0.022	0.022

### *Lead Contents of Furnace Samples*

#### ORIGINAL ANALYSIS

#### CHECK ANALYSIS

0.012

0.005

0.017

0.008

0.018

0.017

0.022

0.020

0.027

0.038

As might be expected, the routine analyses, which are often rushed, do not show the accuracy of the special analyses. From the results given, and from an extended investigation of the electrolytic method in our analytical laboratory by others, it appears that the possible error in the

electrolytic method is of the order of 0.003 per cent. for careful work, and 0.005 per cent. for rapid routine tests.

The readings, in seconds, by the spectroscopic method for the special samples are given in Table 2. These are averages of ten readings by two observers, *A* and *B*. For comparison, the percentage of lead is cal-

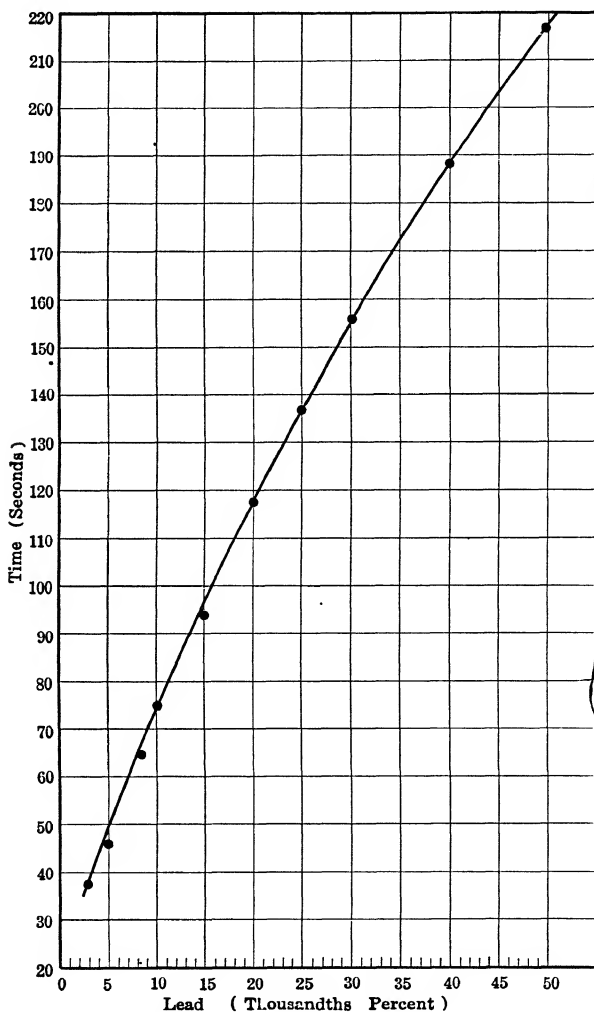


FIG. 4.—CALIBRATION CURVE, ON SYNTHETIC SAMPLES.

(10 amperes, 220 volts. Arc, 1 cm. long. Upper carbon,  $\frac{1}{2}$  in. Lower graphite,  $1\frac{1}{8}$  in.)

culated from the time and the time-lead curve, after this had been established with synthetic samples. The errors expressed are the average deviation from the mean, and would probably represent the order of the possible error.

TABLE 2.—*Comparison of Spectroscopic and Analytic Methods*

No.	Spectroscopic Method				Per Cent. Lead by Analysis	Difference
	Time in Seconds, 2 Observers			Per Cent. Lead from Curve		
	A	B	Av.			
1	65 ± 4	76 ± 7	70 ± 5	0.009 ± 2	0.005 ± 4	0.004
2	107 ± 3	98 ± 5	103 ± 4	0.017 ± 2	0.009 ± 4	0.008
3	94 ± 9	104 ± 7	99 ± 8	0.016 ± 2	0.012 ± 3	0.004
4	88 ± 4	90 ± 8	89 ± 4	0.014 ± 2	0.014 ± 2	0.000
5	122 ± 2	125 ± 8	124 ± 4	0.021 ± 2	0.021 ± 3	0.000

It is to be noted that we have been dealing with the accidental errors of the two methods, and that we have not attempted to express the probable error. The figures indicated the existence of systematic errors, since the determinations by the two methods differ by more than the possible error of each. These differences are the greatest with samples of small lead content, the electrolytic method being uniformly lower than the spectroscopic. It is known that the electrolytic precipitation of the oxidized lead is not complete under certain conditions, and the systematic error would tend to give low results. If we select the highest individual chemical analysis we have a much better agreement between the two methods. Since the spectroscopic method is standardized under the conditions of operation, any systematic errors in the method will be included in the standardization, and we are therefore concerned only with the accidental errors.

### *Synthetic Samples*

Synthetic samples were made in the laboratory by adding a carefully weighed amount of pure lead to the required amount of molten pure copper (Cu 99.963 per cent.) in new fire-clay crucibles under charcoal cover, mixing thoroughly and pouring into fine shot.

The spectroscopic readings were made by the furnace foreman who will use the method in control of the refining furnace. It should be noted that this observer is not experienced in the use of the spectroscope and his training has been in factory production rather than in investigative work. The readings were taken at random, so the observer could have no indication of the lead content of the sample under test. The individual readings are given in Table 3, and from these the averages and errors are calculated. The time-lead curve arising from these readings is shown in Fig. 4. (In regular factory operation the known samples will be run at regular intervals so that any incorrect adjustments will be detected.) This is clearly the best method for standardizing the apparatus.



TABLE 3.—*Time (Seconds) on Synthetic Samples*

Per Cent. Lead	0.000	0.003	0.005	0.008	0.010	0.015	0.022	0.025	0.030	0.040	0.050
Time, sec. ....	0	27	45	56	60	87	122	142	179	203	229
	0	50	30	59	70	111	125	149	171	193	205
	0	50	53	55	94	90	104	139	170	201	207
	0	43	44	51	66	80	115	130	161	160	205
	0	30	45	75	93	86	111	156	138	180	236
	..	38	37	71	78	98	116	132	138	208	
	..	34	44	66	79	102	117	123	158	168	
	..	26	68	67	70	134	112	...	127	191	
	..	..	..	71	..	120	...	...	...	212	
Average.....	..	38	46	65	76	94	117	138	156	188	216
Av. deviation .....	..	9	7	7	8	9	5	9	18	14	13
Prob. error .....	..	3	4	2	3	3	2	3	9	14	6

*High-lead Samples.*—For the sake of completeness, a few tests were run on samples with lead content much above that usually determined in copper refining; results are given in Table 4. On account of the smaller number of observations, the probable error is greater than in the samples of small lead content.

TABLE 4.—*Readings on High-lead Samples*

Per Cent. Lead	0.100	0.150	0.200	0.300
Time, sec . . . . .	240	260	290	408
	228	250	352	456
	245	226	292	450
	222	269	345	448
	248	330	319	437
Average.....	236	267	320	440
Av. deviation.....	8	29	22	14
Prob. error.....	4	12	10	6

Doubt may naturally arise as to possible loss of lead in making the synthetic samples. John Johnston<sup>2</sup> gives the vapor pressure of lead at 1176° C., according to Greenwood's experiments, as 0.022 atmosphere, or 16.7 mm. Hg. Using the equation by Johnston for calculating the vapor pressure, we obtain 15.2 mm. for the same temperature. It is certain that our samples were not heated so high as this and as they were made rapidly it is obvious that the loss of lead must have been slight. The vapor pressure of lead dissolved in molten copper would be less than that of lead alone, at the same temperatures. The close agreement of the analyses by the two methods on special samples No. 4

<sup>1</sup> *Journal Industrial & Engineering Chemistry* (1917) 9, 873.

and 5 would indicate no loss of lead in preparing the samples for standardization.

### VARIATIONS IN CONDITIONS

The variables which might affect the accuracy of the method are:

1. Size of sample.
2. Uniformity of sample.
3. Current variations.
4. Variations in optical apparatus.
5. Arc length.
6. Drafts around electrode.
7. Speed of rotation of electrode.
8. Personal factor.

1-2. *Size and Uniformity of Sample.*—It is obvious that the size of the sample is so small that the method cannot be used with material which is not homogeneous. Enlarging the sample to 1 gm. would hardly make the method applicable to this kind of material. If the furnace sample is properly taken, and is stirred before pouring into shot, we find no evidence of segregation of lead with the low-lead samples which we have been using.

Error is possible in weighing out the sample. Where analytic balances of the usual type are used it is an easy matter to weigh rapidly to 0.001 gm. From the experiments with samples of different weights discussed in our previous paper, it is seen that the time of disappearance of a given unit of lead is not proportional to the weight of the sample. These experiments indicate that an error in weighing of 0.01 gm. would be necessary before the resulting determination of lead would be changed 0.001 per cent.

However, to be certain in this matter, samples having different weights were run, using one sample of low-lead content (0.005 per cent.), and one in which it is quite high (0.050 per cent.). Results are given in Table 5.

TABLE 5.—*Variation due to Weight of Sample*

Weight, Gm.	0.005 Per Cent. Pb., Sec.	0.050 Per Cent. Pb., Sec.
0.397	42	205
0.398	51	
0.399	48	200
0.400	46	210
0.400	54	230
0.401	43	220
0.402	43	211
0.403	40	220
	—	—
Average.....	46	214
Standard (0.400).....	46	216

It is obvious that the small errors in weighing do not affect the lead determination. A systematic error in weighing would affect the standard samples with which the apparatus is checked from time to time, and would soon be detected.

Using the Joly balance, the 0.4-gm. point of which was 45.0 on the scale, the following times were obtained with a sample containing 0.010 per cent. lead, the standard time of which was 75 sec.

READING, JOLY BALANCE	TIME IN SECONDS
44.5	85 79 95
45.0	93 75
45.5	67 76 50 60
Average	75 sec.

The Joly balance can be read within 0.05; hence the error in weighing with the balance will not influence the results.

3. *Current Variations.*—The arc is somewhat unstable, but if sufficient series resistance is included on a high-voltage line, the variations in the arc, when set, are not sufficient to affect the determination. The current should be large enough to melt the sample readily and to keep the globule of copper at a uniform temperature. With low amperages, part of the bead is cold and erratic results are obtained. High amperage increases sputtering but gives better light. While our apparatus is standardized for 10 amp. at 220 volts, good results were obtained with 11 amp. With our apparatus the amperage fluctuates regularly from 9.75 to 10.25, and gives a good average of 10 amp., as shown in Table 6.

TABLE 6.—*Variation due to Fluctuating Current*

Current, Amp.	0.005 Per Cent. Lead, Sec.	0.010 Per Cent. Lead, Sec
8	48 54	80 87
9	43	75
10 (Normal)	42 46	75 76
11	41 40	66 64
12	40 38	58 58
Standard.....	46	75

It would appear that the current variation gives rise to an error of the first magnitude. However, with proper arrangement, the variation can be reduced to a range that will give accurate results.

4. *Variation in Optical Apparatus.*—There can be no disarrangement of the optical apparatus which can affect its accuracy unless the operator attempts to observe the disappearance of small amounts of lead with an apparatus so poorly adjusted that the end is not distinct. One becomes accustomed to the proper setting and immediately notices any change in adjustment.

5. *Arc Length*.—While it is an easy matter to maintain a constant arc length, it is to be remembered that this will not affect the current.

6. *Drafts*.—The arc should not be subjected to drafts of air, since these make the arc unsteady and cool the bead. While we have never attempted to shield the arc, it is quite possible that a properly devised shield would promote even greater accuracy.

7. *Speed of Electrode*.—The speed of rotation of the electrode should be such as to center the arc and keep it from wandering. Too great a speed tends to drafts and sputtering of the arc. In our apparatus the rate is 1000 revolutions per minute.

8. *Personal Factor*.—There appears to be a personal factor in using the method, but it is usually small and constant. One observer may read a certain number of seconds longer than another on the same sample; this is probably due to difference in the sensitivity of the eye. With experienced observers, and even with those who are new to the work, it has been possible for two observers to check each other months apart. Where there is more than one operator it would be safest for each to establish his own calibration curve, using the same standard samples.

#### ADVANTAGES AND LIMITATIONS

It is obvious that the method can be used only on copper in which the lead is uniformly distributed throughout the samples. If care is taken in preparing the shot from the ladle, we have found no evidence of segregation. The occasional readings which vary widely from the average are, in all probability, due to arc wandering or unsteadiness, affecting the temperature of the molten copper. It is our experience that these exceptional readings are almost always high, and we have usually been able to associate such readings with irregularities in the arc. There is a tendency for small particles of the sample to be thrown out from the molten bead; this can be lessened by rotating the upper electrode more slowly and by cutting down the arc current. When reduced to a minimum it does not interfere with the test, but is accounted for in the calibration of the apparatus.

The advantages of the method are facility and accuracy. The analyses can be made in the refining plant by the foreman or by some one who has at least a High School education. The process of refining can be followed rapidly, without holding the furnace for a chemical analysis. Even the last analysis, just before pouring, which requires the greatest accuracy, can be made in a few minutes, and the accuracy of the method at this point is greater than that of the chemical method.

We are indebted to Messrs. Hineline and Nuzum for the spectroscopic tests on the special and synthetic samples, and to Messrs. Gwaltney and Demler for the chemical analyses.

## APPENDICES

1. *List of Materials**Arc Apparatus:*

Motor-generator set.

Westinghouse 3-hp. ind. motor, 220-volt, 3-phase, style 170,906.

Westinghouse 3-hp. d.-c. generator, 230-volt, style 212,677.

Rheostat on motor-generator set, Westinghouse field rheostat, 30 ohms.

Rheostat in series with arc, Westinghouse field rheostat, 200 ohms.  
250-volt style.

$\frac{1}{4}$ -hp. motor, or smaller, for rotating electrode.

3-pole, single-throw, switch for motor-generator set.

2-pole, single-throw, switch for arc and motor.

1 snap switch for motor on rotating electrode.

Ammeter, d.-c., 0 to 15 amp., switchboard type.

Arc adjusting outfit.

Graphite cups and upper carbon electrodes.

Remodeled drill press for rotating upper electrode.

*Optical Apparatus:*

Spectroscope or grating, with lens to focus arc image on slit.

*Accessories:*

Scale, or Joly balance.

Screen for observing arc (colored glass).

Stop-watch.

Standard weight.

2. *Method of Chemical Analysis for Lead in Copper*

A 10-gm. sample is dissolved in 45 c.c. nitric acid (s.g. 1.40). The solution is boiled until evolution of nitric oxide fumes ceases, and it is then neutralized with ammonia. The precipitate is dissolved in nitric acid and 5 c.c. conc. acid is added in excess. The solution is then diluted to 125 c.c. and electrolyzed 30 min. with a current of 1 amp. at 25 volts. The electrode is a platinum gauze cylinder 2 in. high,  $\frac{3}{8}$  in. diameter, with 10-mil wire, 50 to the inch, giving an anodic area of approximately 15 sq. in. or 100 sq. cm. The electrode is washed in alcohol and dried at 180° C.

3. *Test of Joly Balance*

Instead of the regular analytic balance, it has been proposed to use a Joly balance, which may be calibrated from time to time with a standard weight (0.4 gm.) equal to the copper sample to be used. For testing our Joly balance, the weights were  $0.4000 \pm 5$  gm., and the readings (scale in centimeters) were:

44.75	44.80
.62	.60
.75	.75
.70	.75
.75	.78
.80	.75
.80	.75
.80	.75
.80	.75
.75	Average 44.75 $\pm$ 1

Repeated readings with the standard weight on the Joly balance gave the same values ( $44.75 \pm 1$ ). The Joly balance can be made equal if not superior in accuracy to the analytical balance over a given range, and is more rapid. Probably the accurate types of torsion balance, such as made by the Roller Smith Co., Bethlehem, Pa., and used for weighing lamp filaments, might be used in this work, but they are rather expensive.

## Volatilization of Cuprous Chloride on Melting Copper Containing Chlorine

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(New York Meeting, February, 1919)

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### PURPOSE OF INVESTIGATION

Since cuprous chloride melts at  $418^{\circ}\text{C.}$ , boils at  $954^{\circ}\text{C.}$  to  $1032^{\circ}\text{C.}$ ,<sup>1</sup> and is known to be volatile at a much lower temperature, the presence of chlorine in any form in or on copper to be melted has always been looked upon by copper refiners as a possible source of serious copper loss. Although this fact has been known for some time, the copper literature contains little or no information regarding it and the field seems never to have been properly investigated from a metallurgical standpoint.

According to Greenawalt,<sup>2</sup> cupric chloride,  $\text{CuCl}_2$ , when ignited gives cuprous chloride; therefore cuprous chloride is always formed when copper enters into reaction with chlorine at a high temperature. Cuprous chloride melts somewhat below a dull red heat, and does not volatilize in closed vessels, even if strongly heated, but if heated in the air it goes off as a white vapor. Hofman<sup>3</sup> gives the melting point of cuprous chloride

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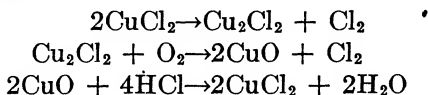
<sup>1</sup> Van Nostrand's *Chemical Annual*, 162. New York, 1918. D. Van Nostrand Co.

<sup>2</sup> W. E. Greenawalt: "Hydrometallurgy of Copper," 163. New York, 1912. McGraw-Hill Book Co.

<sup>3</sup> H. O. Hofman: "Metallurgy of Copper," 57. New York, 1914. McGraw-Hill Book Co.

as 434° C. and states that it is volatile at 340° C. Prescott and Johnson<sup>4</sup> quote Weltzien,<sup>5</sup> that if dry hydrochloric-acid gas be passed over heated copper, cuprous chloride is formed with the evolution of hydrogen.

The reactions of cupric chloride and cuprous chloride at low temperatures have been carefully studied, since they form the basis of the Deacon process for the recovery of chlorine. The reactions involved in that process are as follows:



These reactions are all reversible and large excesses of hydrochloric acid and oxygen are needed to make the reactions occur in the proper direction. In melting copper cathodes containing chlorides, however, the reverse is the case and the proportion of copper to chlorine is so large that, even if cuprous chloride did decompose with the formation of the oxide, the chlorine liberated should at once reform cuprous chloride because of the large excess of copper present.

Samples of flue dust taken from the outgoing gases at the stack of the refining furnaces melting copper cathodes containing such a small trace of chlorine as to be undeterminable have shown that 15 per cent. of the total copper loss at that point was present as cuprous chloride. While the percentage loss was large, the average actual loss was small, amounting to 40 lb. (18 kg.) of cuprous chloride per charge or an equivalent of 0.10 lb. (0.045 kg.) of copper per ton of copper melted. This loss was not large enough to warrant the installation of a costly recovery system, but emphasized the facts of a metallurgical copper loss due to chlorides and the possibilities of much larger stack losses on melting copper containing determinable quantities of chlorine.

On melting copper known to contain chlorine, the fumes given off while charging the furnace have at times so inconvenienced the furnace men as to cause improper charging, materially cutting down the size of the charge and causing labor dissatisfaction. Fumes are also noticed in considerable amount when skimming a furnace charge containing a high percentage of chlorine, and occasionally the fumes from the furnaces are so dense and penetrating as to render it necessary for the workmen to vacate the casting building.

Furnace tests conducted some years ago on melting cement copper produced by the Henderson & Longmaid process, and containing 0.50 per cent. of chlorine, showed a large loss of copper by volatilization as cuprous chloride. The tests, however, were not quantitative and no data

<sup>4</sup> A. B. Prescott and O. C. Johnson: "Qualitative Analysis," 105. New York, 1916. D. Van Nostrand Co.

<sup>5</sup> M. C. Weltzien: *Annales de Chimie et de Physique*, Ser. 4 (1865) 6, 487.



were obtained as to the actual furnace losses. With the recent development of the hydro-electric treatment of copper ores, the cathode tonnage of which is now considerable and continually increasing, the problem of the volatilization of cuprous chloride, if present on the cathodes, is very important owing to the large factors involved. If all the chlorine present on the cathode sheets is considered volatile as cuprous chloride, for every pound of chlorine present on or in the cathodes, a theoretical equivalent of 1.79 lb. of copper will be volatilized during the melting, or an equivalent loss of 3.6 lb. of copper per ton of material for each 0.1 per cent. of chlorine present in the cathodes. It can thus be seen that, where large tonnages are involved, a serious metal loss may result on melting cathode sheets containing but a small percentage of chlorine. In the hydro-electric treatment of copper ores, the presence of chlorides in any form, either in the ore to be leached or in the water used in leaching, will result, on electrolysis, in a deposition of cuprous chloride on the cathodes unless the chlorine is previously removed.

In laboratory experiments, electrolyzing copper sulfate solution containing sodium chloride and using soluble anodes, a white deposit was obtained on the anode, which turned light green in color on drying at 90° C. and had the following composition: Copper 61.84 per cent., chlorine 33.60 per cent.; factor of copper to chlorine, 1.84. On standing 24 hr., the color of the compound changed to a much darker green and had considerably hydrated, as shown by the following analysis: Copper 50.00 per cent., chlorine 26.96 per cent.; factor of copper to chlorine, 1.85.

It is evident that the white deposit was cuprous chloride, which readily became hydrated on 24 hr. standing. A comparison of the two factors shows that there was no oxidation to cupric chloride on standing 24 hr. Duplicating the experiment using lead anodes, the cuprous chloride did not form on the anode but permeated the cathode deposit to such an extent that it could not be readily separated from the deposited copper.

Indirectly, it is easy to prove that a certain percentage of the copper present as cuprous chloride on cathodes is volatilized on melting. Crucible tests made by melting cathode copper containing cuprous chloride always show a copper loss as compared with melting an equivalent weight of cathodes free from chlorides, but the problem is generally complicated by the presence of oxides and moisture on the hydro-electric cathodes and no definite conclusions can be drawn as to just how much copper is volatilized as compared with the original chlorine contents of the cathodes.

The direct method of investigation followed in these experiments consisted in melting 25 gm. of copper, containing chlorides, in an alundum boat and condensing the cuprous chloride volatilized. This cuprous chloride could not be weighed, but was dissolved in water and nitric acid and the copper and chlorine determined by the usual analytic methods.

## DETAILS OF EXPERIMENT

The accompanying illustration shows the apparatus used in these experiments. The copper was weighed and transferred to alundum boats which, in turn, were placed in a 1-in. fused silica tube and the copper melted or heated under a current of carbon dioxide, air, or furnace gases. The carbon dioxide was generated by the action of hydrochloric acid on calcium carbonate and was purified by passing successively through solutions of sulfuric acid, silver nitrate, and sulfuric acid. A No. 17 Fletcher tube furnace with blast was used. No trouble was experienced in melting copper satisfactorily.

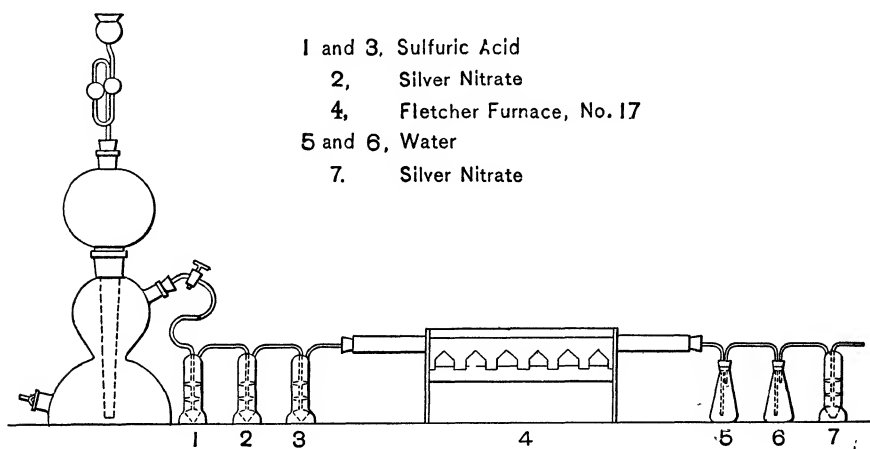


FIG. 1.—APPARATUS USED IN EXPERIMENTS.

The volatilized cuprous chloride condensed partly in the cold end of the silica tube and the remainder was caught by passing the gases successively through two Erlenmeyer flasks containing water, and a Bowen bulb tube containing an acid solution of silver nitrate. After the copper had melted, or had been heated for the specified length of time, the furnace was allowed to cool, the alundum boat removed, and the copper analyzed for any chlorine that was not driven off. The silica tube was carefully washed with water and nitric acid until tests showed the washings to be free from both copper and chlorine. The contents of the Erlenmeyer flasks and Bowen bulb, as well as the washings of all the glass connections, were added to the solution obtained from the silica tube and the total copper and chlorine present determined. The chlorine and copper determinations were made by the same analyst under exactly the same conditions for all the experiments and working independently from the experimenter.

The first series of experiments was conducted while melting hydro-electric copper drillings under a neutral gas, to avoid any possible wrong conclusions, caused by the interference of either an oxidizing or reducing atmosphere. As these experiments were conducted not only to study the volatilization of cuprous chloride, but to arrive at some definite conclusion as to the furnace loss in melting hydro-electric cathode sheets, containing chlorine, under present refining conditions, two other series of experiments were conducted, one aspirating air over the sample while melting, and the other aspirating gases collected from a refining furnace during the melting-down period.

#### EXPERIMENT SERIES No. 1

##### *Melting Copper Containing Chlorine Under Carbon Dioxide*

Owing to the possibility of a very finely divided copper rain being formed as the copper was melting, blanks were run using cast copper, free from chlorine. The heating and melting of this copper and the cleaning of the silica tube and flasks and solution bulbs were conducted in identically the same manner as with the regular experiments. The blank tests were made by melting 25 gm. of drillings of cast copper that was 99.95 per cent. pure and contained no chlorine; the time of heating and melting was 1 hr. The results are given in Table 1. The average loss of copper is small and well within the limit of experimental error and has no influence on the final results and summary.

TABLE 1.—*Melting Drillings of Cast Copper*

Test No.	Weight of Copper in Tube and Bulbs, Gram	Weight of Chlorine in Tube and Bulbs Gram
1	0.0003	nil
2	0.0002	nil
3	0.0004	nil
Average.....	0.0003	nil

##### *Melting Hydro-electric Cathodes*

A carefully prepared representative sample of hydro-electric cathodes was used in this experiment. Analyses showed it to contain 99.58 per cent. copper and 0.226 per cent. chlorine; 25 gm. were used for all tests; and the equivalent weight of chlorine in each test was 0.0565 gm. The time of heating and melting was 1 hr.; 14 tests were made.

The results are given in Table 2. Column 2 gives the weight of chlorine left in the copper after melting. With the exception of test No. 7, this amount is practically negligible. Columns 3 and 4 give the weights of the chlorine and copper that volatilized during the experiment and were determined by washing out the condensed cuprous chloride from tube and flasks. Column 5 shows the ratio of volatile copper to volatile chlorine; that is, simply the ratio of column 4 to column 3. The average factor obtained, 1.76 parts of volatile copper to 1 part of volatile chlorine, is remarkably close to 1.79, the theoretical factor of

TABLE 2.—*Volatilization of Copper and Chlorine on Melting Cathodes*

Test No.	Weight of Chlorine Remaining in Melted Copper, Gram	Weight of Volatile Chlorine, Gram	Weight of Volatile Copper, Gram	Factor Volatile Copper to Volatile Chlorine	Factor Volatile Copper to Chlorine Contents of Sample
1	2	3	4	5	6
1	0.0006	0.0496	0.0820	1.65	1.47
2	0.0006	0.0538	0.0970	1.81	1.74
3	0.0002	0.0497	0.0831	1.67	1.48
4	0.0004	0.0523	0.1011	1.93	1.80
5	0.0009	0.0523	0.0862	1.65	1.55
6	0.0004	0.0546	0.0871	1.60	1.55
7	0.0021	0.0493	0.0861	1.75	1.58
8	0.0003	0.0462	0.0886	1.92	1.58
9	0.0003	0.0515	0.0950	1.85	1.69
10	0.0004	0.0558	0.0970	1.74	1.73
11	0.0004	0.0513	0.0906	1.77	1.61
12	0.0003	0.0516	0.0877	1.70	1.56
13	nil	0.0493	0.0873	1.77	1.55
14	0.0003	0.0575	0.1019	1.77	1.81
Average...	0.0005	0.0518	0.0908	1.76	1.62

copper to chlorine in cuprous chloride, and proves that the chlorine present in the cathodes is volatile as cuprous chloride. Column 6 is the ratio between the copper volatilized, as shown in column 4, to the chlorine contents of the original drillings as shown by the analysis, 0.226 per cent. chlorine or 0.0565 gm. chlorine present in the experiment; due allowance is made for the chlorine left in the melted copper. The average factor 1.62 is based altogether on the analysis of the sample and does not take into account any variation of the chlorine contents in the sample itself. Individual analyses of the same sample show a variation as great as 0.02 per cent. chlorine, which would account for the difference in the two factors.

*Tests on Heating Hydro-electric Cathodes*

In crucible melting tests, it was found that soon after the crucibles containing hydro-electric cathode copper had been placed in the furnace, heavy white fumes were driven off. This continued for some time but before the crucible contents had actually melted these fumes had disappeared, indicating that most of the cuprous chloride was volatilized before the melting temperature of the copper had been reached. In order to learn just what effect heating of the cathodes without melting would have, the following test was made. This experiment was conducted in identically the same manner as the preceding one, except that the copper was not melted, but the drillings were heated at a red heat for 2 hr. The copper and chlorine contents, etc., of the sample were the same as in the preceding experiment. The results of the three tests

TABLE 3.—*Results of Heating Hydro-electric Cathodes*

Test No. 1	Weight of Chlorine Remaining in Copper, Gram 2	Weight of Volatile Chlorine, Gram 3	Weight of Volatile Copper, Gram 4	Factor Volatile Copper to Volatile Chlorine 5	Factor Volatile Copper to Chlorine Con- tents of Sample 6
1	0.0087	0.0487	0.0834	1.71	1.74
2	0.0061	0.0455	0.0824	1.81	1.64
3	0.0090	0.0465	0.0823	1.77	1.73
Average....	0.0079	0.0469	0.0827	1.76	1.70

check closely with those obtained by melting the cathode drillings and again prove the volatilization of copper as cuprous chloride. Column 6, of Table 3, allows for the cuprous chloride remaining in the copper after heating; it is the ratio of the volatile copper to the chlorine contents of the cathodes by analysis, less the chlorine remaining in the copper, as shown in column 2. This factor, based on the analysis of the original drillings, is 1.70 against a factor of 1.76 obtained by comparing the actual volatile copper and volatile chlorine.

*Melting Drillings of Copper Mixed with Sodium Chloride*

It occasionally happens that the chlorine in the cathodes is not present as cuprous chloride, but may be there as sodium chloride. In order to study the effect of sodium chloride on copper, cast copper, 99.95 per cent. pure, free from chlorine was mixed with varying amounts of salt and melted as in the preceding experiments. The salt contains 58.48 per cent. chlorine.

TABLE 4.—*Melting Copper Drillings with Addition of Salt*

Test No.	Weight of Salt Added, Gram	Equivalent of Chlorine Added, Gram	Equivalent of Chlorine in Copper, Per Cent.	Weight of Copper Volatilized, Gram	Factor Volatile Copper to Chlorine Added	Percentage of Chlorine Volatilized as Cuprous Chloride
1	0.5000	0.2924	1.169	0.3259	1.12	62.6
2	0.2500	0.1462	0.584	0.1957	1.34	74.9
3	0.1000	0.0585	0.234	0.1060	1.81	100.0

The results of these tests, given in Table 4, show that the chlorine does not necessarily have to be in combination with the copper to cause volatilization of copper while melting. With an excess of sodium chloride, as in tests 1 and 2, some of the chlorine was volatilized as sodium chloride; but with the addition of small amounts of sodium chloride, approximating the chlorine contents of hydro-electric copper, as in test 3, the chlorine was quantitatively converted and volatilized as cuprous chloride.

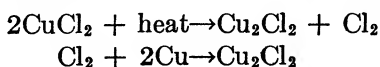
*Melting Cast Copper Drillings Mixed With Cupric Chloride*

Owing to the possibility of the cuprous chloride on the cathodes becoming partly oxidized to the cupric condition on long standing, tests were made by mixing cast copper with cupric chloride and melting the copper as in previous experiments. The cupric chloride contained 37.18 per cent. copper and 41.3 per cent. chlorine; the factor of copper to chlorine was 0.9. While 0.0929 gm. of copper was added as cupric chloride,

TABLE 5.—*Melting Copper Drillings Mixed with Cupric Chloride*

Test No.	Amount of Cupric Chloride Added, Gram	Amount of Copper as Cupric Chloride, Gram	Amount of Chlorine Volatilized, Gram	Amount of Copper Volatilized, Gram	Ratio Volatile Copper to Volatile Chlorine
1	0.250	0.0929	0.0962	0.1742	1.82
2	0.250	0.0929	0.0936	0.1536	1.64
Average....	0.250	0.0929	0.0949	0.1639	1.73

on melting the copper 0.1639 gm. of copper was volatilized with a copper to chlorine factor of 1.73, thus showing that on melting copper containing cupric chloride, the cupric chloride becomes reduced to cuprous chloride and is volatilized as such. Since cupric chloride decomposes on heating to cuprous chloride and chlorine, the reaction must take place in two stages.



## EXPERIMENT SERIES No. 2

*Melting Hydro-electric Cathode Copper Drilling Under Air*

The second series of experiments was made by aspirating air over the copper drillings while melting, thus substituting an oxidizing atmosphere for the neutral atmosphere of the former experiment. The same sample was used as in former experiments, the assay showing 99.58 per cent. copper and 0.226 per cent. chlorine. The time for heating and melting was  $\frac{1}{2}$  hr. The time of heating the drillings had to be shortened, preliminary tests showing that heating the drillings slowly resulted in excessive oxidation of the copper, which prevented the drillings from melting, no matter how much heat was applied. Consequently, a high heat was first applied without any preliminary warming up, in order to melt the copper before the drillings became excessively oxidized. Following this procedure, no difficulty was encountered. The results of the tests are given in Table 6.

TABLE 6.—*Volatilization of Copper and Chlorine on Melting Cathode Drillings Under Air*

Test No. 1	Chlorine Remaining in Melted Copper, Gram 2	Volatile Chlorine, Gram 3	Volatile Copper, Gram 4	Factor Volatile Copper to Volatile Chlorine 5	Factor Volatile Copper to Chlorine Con- tents of Sample 6
1	0.0003	0.0506	0.0880	1.74	1.57
2	0.0005	0.0472	0.0865	1.83	1.54
3	0.0005	0.0607	0.0967	1.59	1.73
4	0.0010	0.0565	0.0920	1.63	1.66
5	0.0005	0.0573	0.1009	1.76	1.80
6	0.0005	0.0560	0.0901	1.61	1.61
Average....	0.0006	0.0547	0.0924	1.69	1.65

Albert M. Smoot, of Ledoux & Co., carrying out an independent investigation on the same sample along similar lines, called attention to the fact of a possible hydrolysis of cuprous chloride and consequent formation of copper oxide should moisture be present in the gases passing over the copper. In order to test this theory, tests 1 and 2 were made with air saturated with water vapor; tests 3 and 4 with dry air; while in tests 5 and 6 the air was neither moistened nor dessicated and represented atmospheric conditions. From Table 6, the average factor of these tests with wet air is 1.56; with dry air, 1.70; and with air at atmospheric conditions, 1.70. These results would tend to prove Mr. Smoot's theory that wet gases would somewhat reduce the volatilization of cuprous chloride. However, furnace gases are neither saturated with water vapor nor are

they dessicated and an average of the six tests should be a fair average of furnace losses allowing for any possible moisture correction.

The factors found agree with those obtained in the former experiments, and show that even if cuprous chloride is oxidized and decomposed the chlorine thus liberated reunites with the copper owing to the mass of the copper present.

### EXPERIMENT SERIES No. 3

#### *Melting Hydro-electric Cathode Drillings Under Furnace Gases*

In order more nearly to approximate actual furnace conditions, furnace gases were collected from refining furnaces during the melting-down period, and tests made on hydro-electric cathode drillings in identically the same manner as in former experiments, aspirating a steady stream of the furnace gases over the drillings while melting. The gas analyses are given in Table 7; the results of the tests, in Table 8.

TABLE 7.—*Analyses of Furnace Gases*

Test No.	CO <sub>2</sub> Per Cent.	O Per Cent.	CO Per Cent.	N Per Cent.
1	10.4	8.2	nil	81.4
2	14.8	2.4	1.0	81.8
3	13.6	5.0	nil	81.4
4	10.4	8.2	nil	81.4
5	15.4	2.8	0.2	81.6
6	11.4	6.2	nil	82.4

TABLE 8.—*Volatilization of Copper and Chlorine on Melting Cathodes Under Furnace Gases*

Test No.	Amount of Chlorine Remaining in Melted Copper, Gram	Amount of Volatile Chlorine, Gram	Amount of Volatile Copper, Gram	Factor Volatile Copper to Volatile Chlorine	Factor Volatile Copper to Chlorine Con- tents of Sample
1	2	3	4	5	6
1	0.0007	0.0479	0.0809	1.69	1.45
2	0.0004	0.0492	0.0842	1.71	1.50
3	0.0003	0.0584	0.1029	1.76	1.83
4	0.0005	0.0588	0.0959	1.63	1.71
5	0.0009	0.0539	0.0861	1.60	1.55
6	0.0008	0.0540	0.0858	1.59	1.54
Average....	0.0006	0.0537	0.0893	1.66	1.60

As in the preceding experiments, tests 1 and 2 were made with the gases saturated with water vapor; tests 3 and 4 with dried gases. In tests 5 and 6, while the gases were neither moistened nor dessicated they



were collected from the furnaces over water and were displaced during the experiment by water, so that the gases used contained more water vapor than under actual furnace condition. From Table 8 the average factor of these tests with wet gases is 1.48; dry gases, 1.77; gases as collected, 1.55.

These results again tend to prove Mr. Smoot's hydrolysis theory. The presence of water vapor in the gases seems to have more effect on the volatilization of cuprous chloride than the composition of gases passed over the copper while melting. The average of the six tests made under the three different conditions should give a fair average of furnace losses, allowing for any possible moisture correction; possibly a little low, because the gases in tests 5 and 6 contain more water vapor than is actually present in the furnace gases.

### SUMMARY

Experiments in melting drillings of cathode copper containing cuprous chloride, and analyzing the resulting volatile copper and chlorine, resulted in the following series of factors:

#### *Factor Volatile Copper to Volatile Chlorine Based on Actual Determination [of Volatile Products]*

Melting under carbon dioxide (14 tests).....	1.76.
Melting under air (6 tests).....	1.69
Melting under furnace gases (6 tests).....	1.66

#### *Factor Volatile Copper to the Actual Chlorine Contents of Sample*

Melting under carbon dioxide.....	1.62
Melting under air.....	1.65
Melting under furnace gases.....	1.60

Considering that the theoretical factor of copper to chlorine in cuprous chloride is 1.79, these experiments prove that the volatilization of cuprous chloride on melting cathode copper takes place almost in its molecular ratio, and that under present copper refining practice any cuprous chloride present in or on the cathode can be considered, for all practical purposes, as completely volatilized on melting, and may be the cause of a serious metallurgical loss of copper.

## Automatic Copper Plating

BY JOSEPH W. RICHARDS,\* SOUTH BETHLEHEM, PA.

(New York Meeting, February, 1919)

PLATING iron with copper has received great attention from practical and scientific men, but, aside from the deposit secured by immersion of iron in copper salts, by electro-plating, or by welding together thin sheets of iron and copper, these efforts have met with no success.

Failure of these efforts, in the production of copper plating, has been due to the fact that the conditions normally and necessarily present in the plating operation are antagonistic to the production of copper-plated iron. In most, if not quite all of them, a bath of molten copper has been used. The temperature of a molten copper bath is so high that the iron becomes oxidized before it can be immersed in the molten copper, and unless a protective flux for the molten copper is used, the surface of the copper will become oxidized, and, in any event, the plated iron will oxidize immediately on its being withdrawn from the molten bath.

I recently had the pleasure of visiting the works of Thé Metals Plating Co. at Elizabeth, N. J., where I witnessed the plating of iron sheets with copper by a new process.<sup>1</sup> The plating metal is applied to the sheet in the form of a liquid mixture by means of rolls, such as inking rolls. The sheet, after being coated with the mixture, is automatically carried forward and deposited on a link-belt conveyor, which carries it through a furnace maintained at a temperature well above that of molten copper. The basic principle involved in this method lies in the application of the plating metal to the sheet while the sheet is cold and then melting the metal in place on the sheet under conditions which are favorable to the formation of the plating.

### PLATING MIXTURE

The liquid plating mixture is composed of either copper oxide or finely divided copper, or a mixture of both, ground to the consistency of a light varnish in a crude oil having an asphaltic base, of a specific gravity of from 14° to 16° Bé. The plating mixture which gives the best results

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\*Professor of Metallurgy, Lehigh University.

<sup>1</sup>U. S. Patents 1197693, 1197694, 1197695, Sept. 12, 1916, to William E Watkins.

consists of 4 lb. (1.8 kg.) of copper oxide ground together with 4 lb. of finely precipitated copper, and made to the consistency of a light varnish by grinding it in 1 gal. (3.8 l.) of Mexican crude oil of specific gravity of 14° to 16° Bé. It is found that the asphaltic base of this oil has reducing power sufficient to reduce the oxide of copper to metal, in the furnace, and to protect the precipitated copper from oxidation during the operation; also to reduce any oxide of iron that may have been on the sheet. One gallon of oil can reduce 5 lb. of copper oxide.

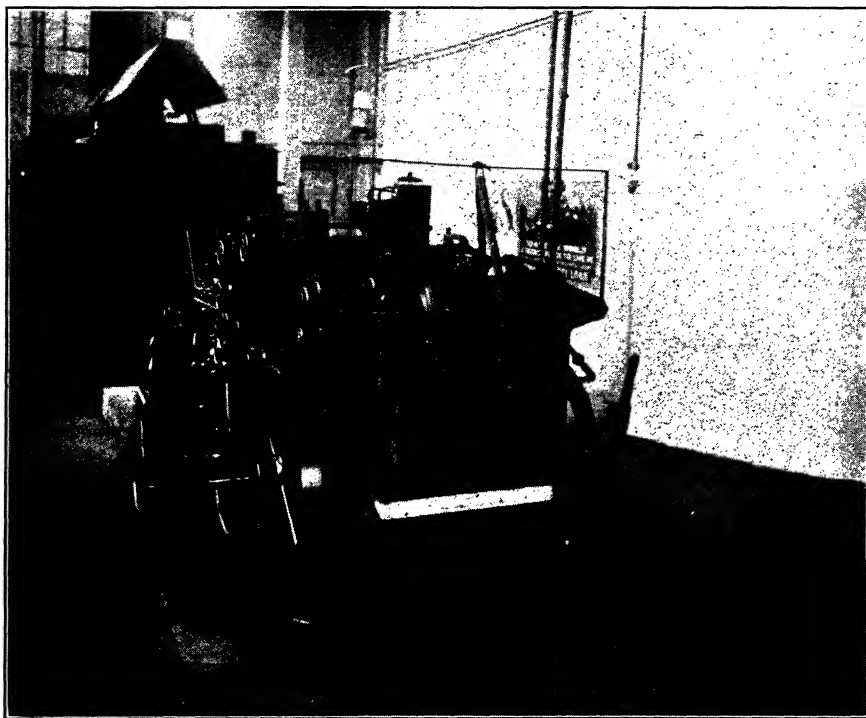


FIG. 1.—SHEET-FEEDING MACHINE AND COATING ROLLS.

This mixture has also been found to have the proper viscosity for its application to the sheet by coating or inking rolls, and to hold it uniformly on the sheet when exposed to the furnace temperature so as to produce a uniform deposition of strongly adherent copper upon the sheet. A number of variations of this mixture have been used, such as using copper oxide only, mixed with powdered charcoal, or using precipitated copper only without any copper compound. Mexican crude oil is probably the cheapest liquid to use, suitable for this purpose, but other liquids of similar properties have been used with nearly equivalent results.

## AUTOMATIC FEEDING AND PLATING MACHINE

As the boxes of sheets are placed on the platform of the machine, it is necessary to lift the sheets one by one and feed them, like feeding paper to a printing press. This can be done by hand but has been accomplished very satisfactorily by an automatic sheet-feeding machine which has been worked out by Mr. Conran, the company's superintendent. The platform on which the stack of sheets is placed is raised continually so that the top sheet is always at the same level. A suction cup comes into contact with the upper sheet and lifts it, while at the same moment a jet

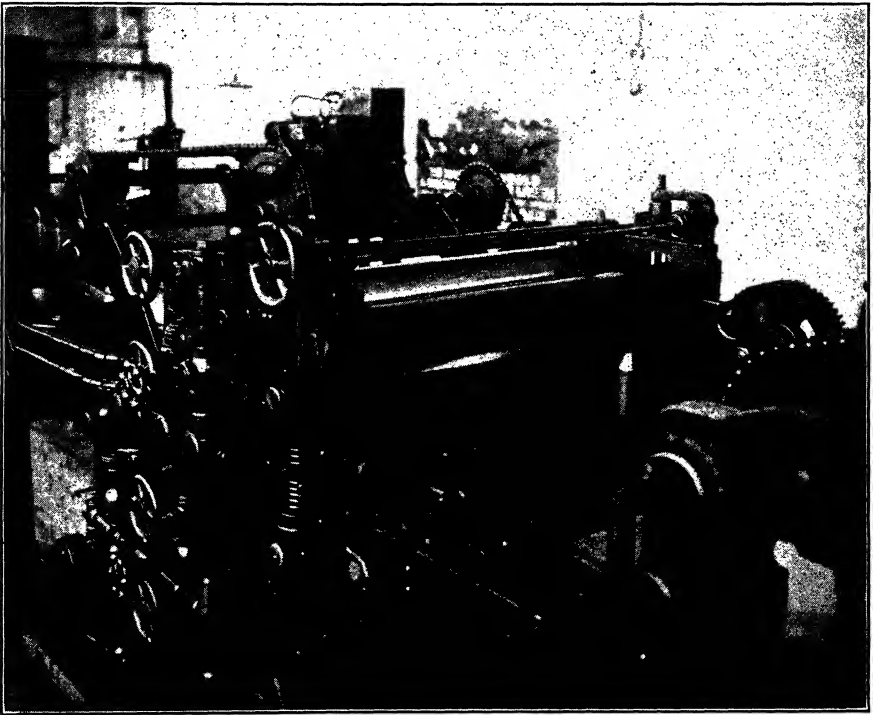


FIG. 2.—COATING ROLLS SHOWING PARALLEL ADJUSTING GEARS.

of compressed air is introduced beneath the sheet to destroy the vacuum effect, or any other slight adherence between the sheets, thus freeing it from the sheet beneath. The suction cup raises the rear end of the sheet and forwards it into engagement with the coating rolls to receive the plating mixture. The sheet then travels forward and is deposited on the furnace conveyor, lateral fingers compressing the sheet into an arch, so that it passes into the furnace bowed, in a position and shape best suited for withstanding collapse when exposed to the heat existing in the furnace.

Parts of the carrier, such as the fingers that support the sheet, that are exposed to the greatest heat of the flame, are cast of nichrome metal, in order that they may withstand the temperature and the oxidizing influences of the flame.

While being carried through the furnace, volatile constituents of the paint upon the sheets distill, copper oxide in it is reduced to metal, and the copper unites with the iron to form a continuous coating. The atmosphere of the furnace is kept reducing in order to preserve the coat-

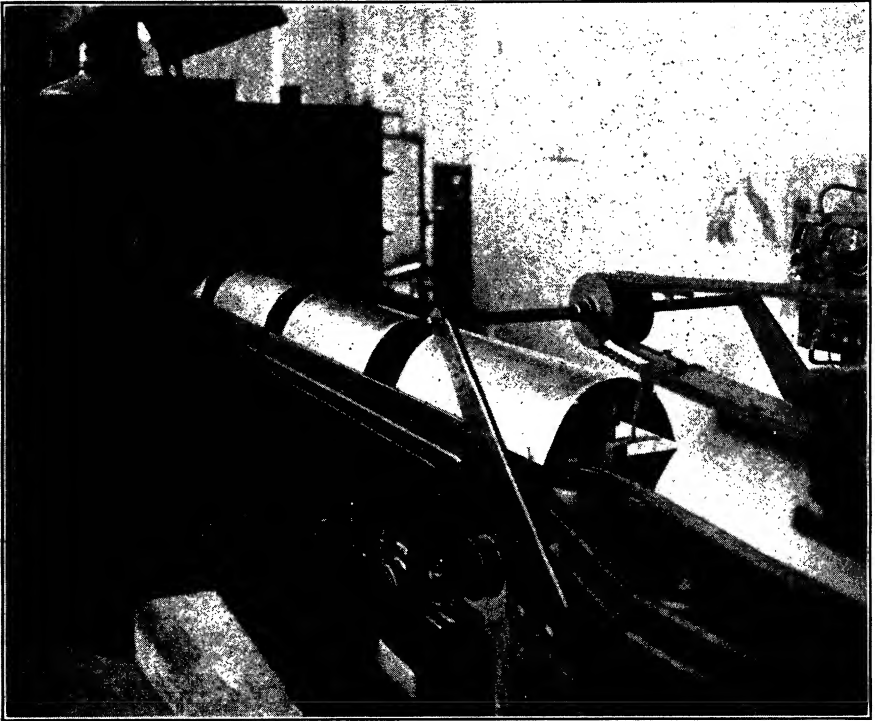


FIG. 3.—SHEET-ARCHING MACHINE AND FURNACE CONVEYOR, SHOWING BOWED SHEETS ON THEIR SUPPORTS ENTERING THE FURNACE.

ing as far as possible from oxidation. The carrier delivers the sheets to flattening rolls, which pass them on to another carrier operating at lower temperature, upon which they cool.

Articles of other shapes than sheets, such as wire and tubes, can likewise be copper plated on the same principle and by the use of similar continuous automatic apparatus. Other metals as well as copper, such as tin, lead, and alloys of different metals, can also be used as the plating metal.

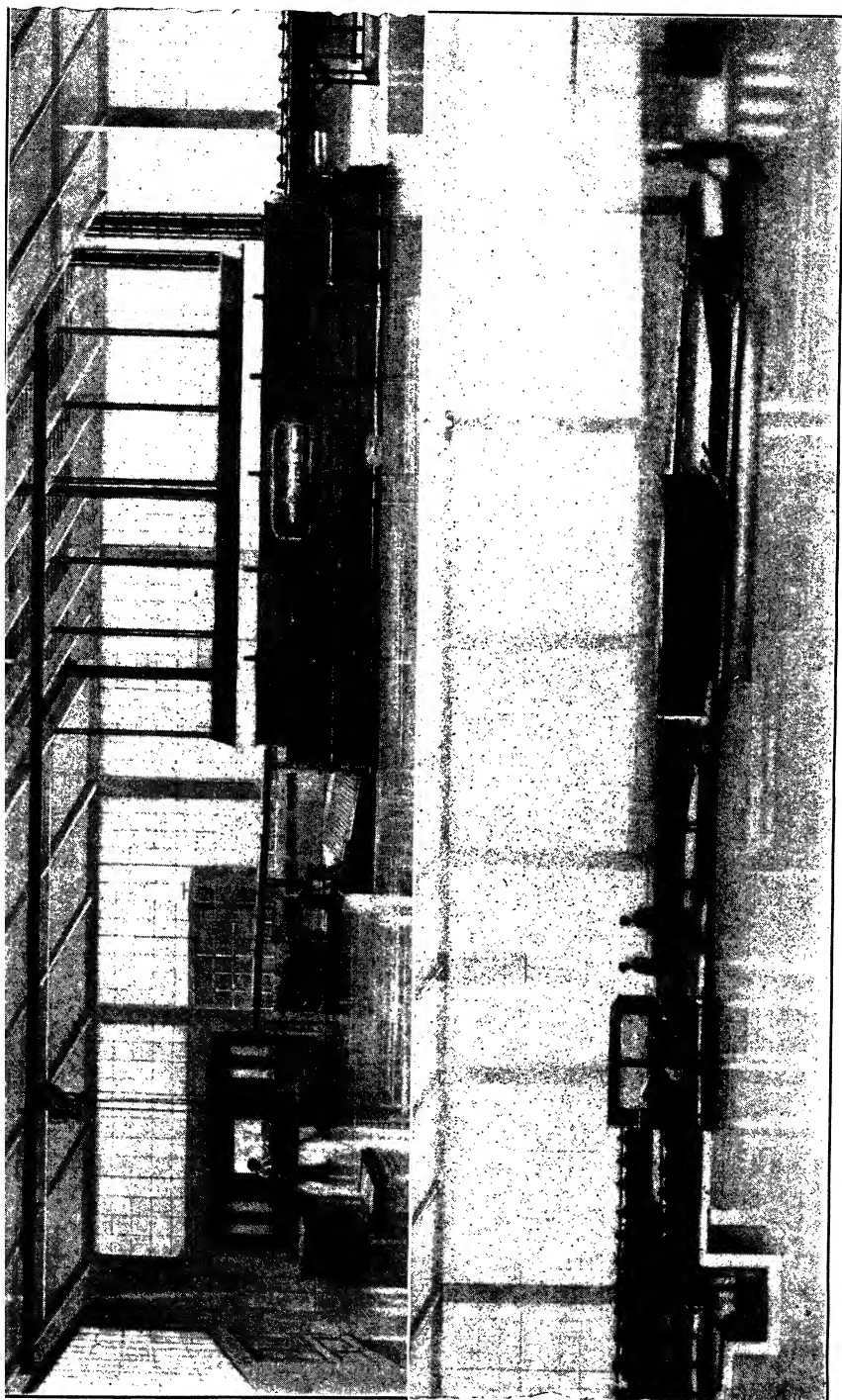


FIG. 4.—AUTOMATIC FEEDING AND PLATING MACHINE.

## DISCUSSION

A. SILVERMAN,\* Pittsburgh, Pa.—Prof. Richards has described a very interesting process. In addition to the method described, electroplating and a number of other processes have been used. In one, the two metals were heated and united under hydraulic pressure; in a second, the steel was coated with an alloy, brass for example, and the molten copper cast around this. The process that I would like to bring to the attention of the metallurgists present consists simply of pouring molten copper, brass, or cupro-nickel around a clean red-hot steel billet. It is covered by the Roth patents. The 8-in. cylindrical billet containing about 70 per cent. steel (core) and 30 per cent. copper is hot drawn or rolled until several inches in diameter and afterward cold drawn or rolled to rod, wire of any desired diameter, or sheet. The wire is used for electrical conduits where conductivity and high tensile strength are required, also for the manufacture of cables and screen cloth. The sheet is employed for the manufacture of spouting, molding, shells, etc.

The last application is particularly interesting. In testing rifle cartridges, which usually contain lead-filled copper noses, pine boards 1 in. thick, covered with  $\frac{1}{4}$ -in. rawhide, are placed 1 ft. apart. The value of the cartridge depends on the number of boards penetrated by the bullet. In Government tests made with copper-coated steel noses (15 per cent. copper on either side of the steel) the penetrating power was found to be approximately double that of pure copper noses. The advantage of copper-coated steel over cupro-nickel lies in the fact that the former does not require any special heat treatment during the punching. Rifle cartridges were prepared from copper-coated steel in a number of Government arsenals. The value of the combination was attested by the awarding of a contract for the construction of a \$3,300,000 plant. Unfortunately, the contract was signed on the day preceding the signing of the armistice and was cancelled several days later. Tests were conducted for about 1 yr. before the contract was awarded to determine the value of this combination of metals. Since the war a number of cartridge and shell manufacturers have signified their intention of using copper-coated steel.

Many other interesting applications of copper, brass, and cupro-nickel-coated steel are possible. Copper-coated steel springs have already been manufactured. Ground pins and connecting wires for rails are in use. Wire 0.005 in. in diameter looks like pure copper or brass depending on the coat, and thin sheet also resembles the pure metal. In fact, were it not for the springiness of the steel filling, one could not tell the coated material from the pure copper or brass. Copper- and brass-coated steel, as their value becomes apparent, will take an important place in the metal industry of the near future.

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\* Head of the School of Chemistry, University of Pittsburgh.

## Pure Carbon-free Manganese and Manganese Copper

BY ARTHUR F. BRAID,\* NEW YORK, N. Y.

(Milwaukee Meeting, October, 1918)

THE war has caused an increasing scarcity of phosphorus and its well known alloys with copper and tin. At the same time, the production of brass and bronze, nickel-silver, cupro-nickel, and other non-ferrous alloys, has considerably increased. The manufacturers of these products had therefore to secure other materials which would serve their purpose; principally that of a deoxidizer, which could be obtained promptly and regularly.

Fortunately these materials, pure carbon-free manganese metal and manganese-copper alloy, were not hard to find—in fact, they were never lost. Manganese in various forms has been used in Europe for more than a century; in this country, however, when manganese was first used, and indeed for a long time thereafter, it was consumed mainly in the manufacture of manganese-bronze. Many foundrymen at that time used manganese-copper with the same freedom as phosphor-copper. Although iron enters into the composition of certain grades of bronze, it is very detrimental to non-ferrous mixtures in general; consequently ferromanganese is not applicable, and therefore pure manganese metal, or the alloy of manganese and copper, technically free from iron and other impurities, is now being generally used.

The following are the principal elements having an affinity for oxygen: 1, Sodium. 2, Potassium. 3, Calcium. 4, Strontium. 5, Barium. 6, Magnesium. 7, Aluminum, 8, Phosphorus. 9, Silicon. 10, Manganese. 11, Iron. 12, Zinc. 13, Lead. Each metal has its natural flux, or deoxidizer, in the form of a metal or non-metal which will alloy with it, and has a strong affinity for oxygen. For example, phosphorus, as is well known, acts very beneficially in copper alloys that contain tin.

Within the last few years, and particularly since the war began, the great value of manganese in nickel alloys has been demonstrated, and one by one the manufacturers of these products have adopted its use. While manganese has not so strong an affinity for oxygen as magnesium, aluminum, or silicon, it is nevertheless sufficiently powerful to reduce any oxide of nickel, copper, or zinc that may be present in the nickel alloy to be purified.

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\* Metallurgical Engineer, Metal and Thermit Corporation.



It is this "medium" affinity for oxygen that renders manganese valuable in casting nickel alloys for rolling or drawing purposes, since the oil, burning at the mouth of the mold, is capable of reducing the thin film of manganese oxide that forms upon the stream of metal as it is poured. Aluminum or silicon oxides are not reduced by burning oil, and, therefore, it frequently happens that castings are dirty when aluminum or silicon is used; with manganese, clean castings result if ordinary precautions are taken in pouring.

It is customary to add the manganese in the form of manganese-copper alloy (30 per cent. Mn and 70 per cent. Cu). The addition to nickel silver is about 3 or 4 oz. of the alloy per 100 lb. of the mixture (2 gm. per kilo), equivalent to about 0.06 to 0.075 per cent. manganese.

In such alloys as cupro-nickel, the amount of manganese-copper should be almost doubled. This quantity introduces about 0.12 to 0.15 per cent. manganese, and to certain grades of nickel-copper, 0.25 per cent. manganese is added. The manganese-copper should be introduced into the mixture after all the other metals have been melted and the whole well stirred. The mixture should then be left for a few minutes so as to give the manganese time to act. It will be found that manganese gives excellent results in nickel alloys.

Another important feature in the use of manganese is its strong affinity for sulfur, exceeding that of all other metals; when introduced into a mixture containing sulfur, sulfide of manganese is formed at once, and rises as slag to the top of the metal. As sulfur is frequently present in cupro-nickel alloys, the advantage of this property of manganese is obvious. I believe that more attention should be given to sulfur in non-ferrous mixtures, particularly at the present time when raw materials and fuel are not generally of so high quality as before the war.

In the casting of brass, it has been shown that manganese acts best when there is a high proportion of zinc. On the other hand, in a red brass, phosphorus is used with excellent results because it causes the tin, with which it alloys more readily, to assume a crystalline structure and produces a more homogeneous casting. A number of concerns are experimenting with manganese, to take advantage of its hardening effect, and thus diminish the amount of tin used in certain mixtures.

The present tremendous increase in the use of manganese has been brought about to a great extent by the demand for munitions, etc., since, in addition to the various mixtures previously mentioned, manganese is used in nichrome, monel metal, aluminum, and stellite, the well known high-speed cutting tool, as well as in other less well known mixtures. Probably the most recent use for manganese is in a certain non-ferrous mixture which is used for the production of the necessary charcoal for gas masks by the carbonization of fruit stones.

## DISCUSSION

W. B. PRICE,\* Waterbury, Conn. (written discussion†).—It has been our experience, especially in cupro-nickel, that manganese has been very beneficial, not only, as stated by Mr. Braid, in removing sulfur, but also for its property of reducing oxides, thus making the metal more fluid, and enabling it to be poured at a lower temperature; this, in turn, prevents softening of the graphite pots and the absorption of carbon. It also acts very effectively in keeping the carbon in solution in the cupro-nickel, in the combined form. However, in the case of cupro-nickel, to obtain all these benefits it has been found necessary to keep the manganese content as near 0.25 per cent. as possible.

Manganese also is very effective in diminishing the amount of blisters in cupro-nickel. So far as I am aware, both from personal experience and from the experience of others who have been engaged in the manufacture of nickel-silver and cupro-nickel, manganese used either in the form of pure metal or as manganese-copper alloy (30 Mn, 70 Cu) gives better results than any other flux. While this may seem a strong statement, manganese is the natural flux for nickel-silver and cupro-nickel.

W. H. BASSETT,‡ Waterbury, Conn.—Manganese should not be expected to remedy all the defects that are due to poor melting practice. It is undoubtedly valuable in helping to eliminate porosity in many of the alloys. In the copper-nickel alloys, its use is becoming almost universal, for, beside helping to make sound castings, it tends to hold the carbon in solution in the solidified alloy.

G. H. CLAMER,§ Philadelphia, Pa.—I have found that manganese copper is satisfactory for eliminating sulfur and that it works very well in the crucible and when you have reducing conditions; but in a large reverberatory charge, where the conditions are oxidizing, we were not able to eliminate the sulfur.

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\* Metallurgist with the Scovill Manufacturing Co.

† Received Sept. 30, 1918.

‡ The American Brass Co.

§ First Vice-president and Secretary, Ajax Metal Co.

### Manganese Bronze\*

BY P. E. MCKINNEY,† WASHINGTON, D. C.

(New York Meeting, February, 1919)

DEVELOPMENTS in engineering during the past decade, particularly as applied to marine construction, mining machinery and other purposes in which corrosion offers a serious problem, have created a large demand for a non-ferrous metal highly resistant to corrosion and at the same time useful in general construction work as a substitute for steel without materially decreasing the factor of safety or increasing the weight of the various parts over that ordinarily used in the case of mild steel.

To obtain the combination of desired properties many compositions have been proposed and used with excellent success, but at the present time probably the most popular and most widely used combination is the non-ferrous alloy commonly called manganese bronze. This is nothing more than a high brass to which have been added, by the proper method of alloying, comparatively small percentages of aluminum, iron, or manganese with the definite purpose in view of strengthening the alloy and rendering it more dense and close-grained than the average yellow-brass casting.

In the manufacture of manganese bronze a great deal has been said about the importance of using only the highest grades of raw material and the beneficial or detrimental effects, as the case may be, of various impurities, as well as the importance of adding the ingredients according to various formulas proposed; but in most of these cases the literature on the subject has dealt principally with the manufacture of this alloy from virgin metals and raw materials of the highest purity. It is the purpose of this paper to deal particularly with the possibilities that lie in the proper development of methods for manufacturing such an alloy by more economical methods than those which have usually been discussed in other literature on the subject. Shortages have recently existed in raw materials needed for many products entering into the various branches of engineering work, particularly the non-ferrous metals, and during the next few years of reconstruction the country will be flooded with many

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\* Published by permission of the Commandant and Superintendent, U. S. Navy Yard, Washington, D. C.

† Chemist and Metallurgist of the Naval Gun Factory, U. S. Navy Yard.

byproducts and much scrap resulting from the extensive operations carried on during the period of the war, during which time scrap and byproducts could not be handled in sufficient volume to effect rapid production. These facts constitute conditions that make a project looking toward the efficient utilization of such materials well worth while.

When manufacturing manganese bronze or similar alloys from raw materials there is intentionally added a percentage of iron, which is generally conceded to be one of the very objectionable impurities in non-ferrous scrap, in addition to which aluminum and manganese are added, and sometimes tin in small quantities, all of which if present in the virgin metals ordinarily used in non-ferrous foundry practice would be considered detrimental impurities. The composition of the average manganese bronze is as follows:

	PER CENT.
Copper . . . . .	57.00 to 59.00
Zinc . . . . .	38.00 to 40.00
Iron, manganese, aluminum, tin. . . . .	0.25 to 1.00
Lead . . . . .	0.10 to 0.50

which composition shows rather clearly that there is no necessity for the use of high-grade raw materials, provided methods of manufacture can be devised to produce the proper refinement of the finished product, as the ordinary impurities encountered in non-ferrous materials offer no serious obstacles when the same elements must be added in considerable percentages to effect the desired composition.

In the manufacture of manganese bronze it has been found possible to utilize what would ordinarily be termed material of very low grade, such as skimmings from the foundry, particularly skimmings and dross ordinarily recovered from brass rolling mills or cartridge-case plants, zinc dross recovered from galvanizing plants, aluminum turnings that are ordinarily unrecoverable without serious loss and deterioration of the product due to oxidation, etc., and other byproducts and scrap metals that ordinarily are not usable as remelting scrap in foundry practice. The manufacture of high-grade manganese bronze from materials of this class, however, cannot be attempted in crucible furnaces, or practiced only on a comparatively small scale, as there is required a reverberatory or other furnace in which it is possible to accumulate a bath of considerable volume and in which the charge of metal can be worked in the same manner as in other refining processes producing on a large scale in open-flame furnaces. The idea is to so compound the various materials available as to cause one to react with the other to the mutual benefit of all the ingredients going into the charge.

A typical charge for operating a reverberatory furnace in the manufacture of manganese bronze is as follows:

	Pounds
Yellow-brass machine-shop turnings.. . . . .	1250
Zinc dross from galvanizing plant.....	400
Aluminum turnings . . . . .	30
Recovered scrap zinc.....	400
Dross and skimmings from billet and slab plant.....	2000
Foundry-floor scrap and skimmings . . . . .	650
Hardener made by melting scrap copper and manganese in equal proportions . . . . .	250
Charcoal.....	75
Common salt . . . . .	80

With a knowledge of the general chemical composition of each ingredient going into the charge it is easy to produce a manganese bronze that will have the desired composition and will require no other treatment than, possibly, the addition of a little zinc when remelting for casting purposes.

When handling a charge such as that just shown, advantage is taken of impurities, such as the iron existing in the zinc dross, the small percentages of tin in the machine-shop turnings, etc., in figuring the final desired analysis of the alloy. It is a well-known fact that such materials as zinc dross and foundry skimmings, both of which are full of dirt and oxides, cannot be recovered in a usable condition when melted separately in crucibles or by any of the ordinary methods of melting material, without an excessive loss due to volatilization. But when the well-known reactions occurring in the manufacture of old-fashioned calamine brass are taken into consideration, it can be seen how the combination of zinc dross, brass skimmings, and other highly oxidized materials melted in contact with charcoal will produce an alloy about as well deoxidized as the average high-zinc brass. Contact, while in a molten or semi-molten state, of metallic copper and partly oxidized zinc in the presence of charcoal develops a condition extremely conducive to a thorough deoxidation and alloying of all the metallic ingredients. This reaction, together with the well-known deoxidizing effects of aluminum, manganese and iron, which are part of the charge, contributes to the production of thoroughly deoxidized and dense material. Salt is used as a flux on account of its excellent properties for gathering and fluxing off the dirt from the charge, the oxides of iron, manganese, etc., and the reduction of any copper oxides passing from the bath into the slag.

In charging the furnace, it is the usual practice to place the manganese hardener in the bottom of the furnace and on top of it place the cartridge-case dross and zinc dross with a layer of charcoal, after which the foundry skimmings and turnings are added and the charge is melted, with occasional rabbling to clear the metal and bring all the ingredients into intimate contact. When the bath is melted, the aluminum scrap is added and the necessary zinc additions are made, after which the charge

is allowed to cool slightly in the furnace and is poured into ingots for remelting purposes, after analysis has been made to determine the necessity of adding zinc or otherwise correcting the composition by the mixture of several heats in order to obtain the desired composition.

The foregoing process carried out by a skilled melter will produce an alloy that is absolutely sound and homogeneous and capable of being cast in ordinary foundry practice in the same manner as any of the so-called high grades of manganese bronze, and will produce most excellent physical properties in the finished casting.

As instances of what can be obtained, the following results of tests are given. These tests were made on castings produced by the method described from initial charges of raw material practically identical with the typical charge given.

TABLE 1.—*Analyses of Ingots as Poured from Reverberatory Furnace*

	Copper, Per Cent.	Zinc, Per Cent.	Tin, Per Cent.	Lead, Per Cent.	Iron, Per Cent.	Aluminum, Per Cent.	Manganese, Per Cent.
Heat A. . . .	57.95	39.08	0.35	0.42	0.83	0.59	0.77
Heat B. . . .	57.18	39.54	0.51	0.34	1.04	0.48	0.91

TABLE 2.—*Physical Values Obtained on Sand Castings Poured from Remelt of Ingots without Additions*

	Tensile Strength, Lb. per Sq. In.	Yield Point, Lb. per Sq. In.	Elongation, Per Cent.
Heat A. . . .	67,700	46,300	22.1
Heat B. . . .	72,100	50,400	21.0

TABLE 3.—*Physical Tests of Sand Castings Made from Manganese Bronze Produced by Process Described*

Tensile Strength, Lb. per Sq. In.	Yield Point, Lb. per Sq. In.	Elongation, Per Cent.
67,300	45,800	29.9
66,400	37,400	30.9
67,700	41,200	24.5
72,200	34,100	24.5
67,900	33,900	28.1
72,600	36,700	23.4

In addition to the excellent properties shown in the tensile tests, the material corresponds in every other physical respect to so-called high-grade manganese bronze produced from virgin metals, has a fine uniform

fracture with a tendency to silky grain in many cases and shows toughness and resiliency equal to any other grades of manganese bronze examined.

Some objections may be raised to the use of byproducts and scrap material in the manufacture of manganese bronze because the lead content of some of these byproducts is quite high and will introduce into the alloy quantities of lead higher than are desired. It is believed, however, that the results of tests made from time to time on manganese bronze, containing in some cases a considerable percentage of lead, have shown conclusively that lead within reasonable limits is not detrimental. No concrete cases have as yet been cited in which, other things being equal, lead under 0.75 per cent. has shown seriously detrimental effects on the alloy. All the samples on which physical tests are given contained percentages of lead ranging from 0.25 to 0.50 per cent., yet none of them show in the test results any detrimental effects due to the presence of this so-called impurity. It is believed that the tolerance of a slightly higher content of lead in specifications for manganese bronze would, in general, serve admirably as a conservation measure for high-grade virgin material and offer excellent opportunities for the utilization of many metallurgical byproducts that at the present time constitute practically useless material.

The results obtained after about 3 years of operation under the process described have shown rather conclusively that the alloy commonly known as manganese bronze can be produced without resorting to the use of high-grade virgin materials with the addition of what would ordinarily be termed detrimental impurities, by the simple application of some of the well-known laws of metallurgy.

## DISCUSSION

JESSE L. JONES,\* Pittsburgh, Pa. (written discussion†).—The most important criterion on which judgment of the quality of manganese bronze can be based is its freedom from lead. Not over 0.03 per cent. of lead should be present in a good grade of manganese bronze. While it is not questioned that manganese-bronze with a high tensile strength and considerable ductility can be made from materials that will give a lead content of possibly 2 to 3 per cent., there is no doubt that such material, while it may machine readily, will be found to withstand the impact test very poorly, and hence be unsuited for high-grade engineering work. When lead is present in considerable amounts, it forms in small pools in the alloy and the material has little strength at these points; in fact, such spots in the metal are equivalent to nicks in a test bar as far as their effect on tensile strength is concerned. A further objection to a high lead content is the ease with which it oxidizes and its consequent tendency to produce dross.

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\* Metallurgist, Westinghouse Elec. & Mfg. Co. † Received Feb. 13, 1919.

A second characteristic of a high-grade manganese bronze is a low content of manganese. This should not exceed 0.05 per cent.; in fact, very many of the best grades of manganese bronze show, on analysis, only a trace of manganese or no manganese whatever. Manganese is a deoxidizer and its ready oxidation protects the other metals of the alloy; but if it is present in large amounts it will produce considerable manganese dioxide, which does not separate readily from the molten alloy and may produce black specks or drossy spots and render the obtaining of clean castings difficult.

Aluminum should be present in a good grade of manganese bronze in only very small amounts. A content of 0.10 per cent. aluminum is ample. Larger amounts have a tendency to increase the shrinkage of the alloy, to give the ingots and castings a white, scruffy appearance, and to prevent the beautiful, golden, oxidation color characteristic of high-grade manganese bronze and a most excellent indication of quality.

The work that the author of the paper has done is valuable from a conservation standpoint, and it will supplement, in a marked degree, the advances that have been made by a number of manufacturers of manganese bronze in utilizing the turnings from this material. A number of firms have succeeded in making a very good grade of ingot metal from turnings and other light scrap, obtaining a tensile strength and elongation which, although not equal to that obtained from new metal, is still very creditable.

The lack of uniformity in manganese bronze made from the materials specified by the author of the paper, the tendency to drossy metal from the extremely high content of lead, manganese, and aluminum, which are shown by the analyses given, combine to render the commercial use of manganese bronze made from such inferior materials of doubtful utility.

There is a field, however, for what might be designated as a second-class quality of manganese bronze in the making of rather light castings. These castings can be gated in such a way that dross and oxide will be strained from the metal and clean castings thus produced. The castings set quickly; hence if the metal has a rather high shrinkage it is not so likely to be objectionable as in a heavier casting. In light castings, also, the oxidation of the metal in the mold cannot occur to any marked extent, as in the case of large molds that are very slowly filled and in which the metal is a long time congealing. Hence, the grade of manganese bronze described by the author can be used for such castings but its use in large and important castings is likely to be far from satisfactory, for the reasons named.

W. M. CORSE,\* Mansfield, Ohio (written discussion†).—Mr. McKinney has called attention to a very practical method of making manga-

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\* Manufacturing Engineer, Ohio Brass Co.

† Received Feb. 17, 1919.



anese bronze and has checked the method by about 3 years of operation. The Naval Gun Factory has an unusual opportunity to check physical results from metal in castings because every heat is tested physically and a splendid series of results is thereby on record. Very few commercial foundries, unless on government work, test every heat of metal physically. As a result, the physical properties published by commercial foundries are not backed by nearly as many tests. I mention this to bring out the fact that Mr. McKinney is not describing an occasional heat of manganese bronze but is telling of results that are checked constantly by physical tests.

The point made that the method of making manganese bronze as described is not applicable to strictly crucible practice is important; also that the method is not practicable with small quantities of metal. The fact that less than 0.75 per cent. lead is not markedly harmful to the physical properties of this alloy is interesting, particularly in view of existing specifications.

HAROLD J. ROAST,\* Montreal, Que.—About 2 years ago Mr. McKinney told me about mixing zinc dross with foundry skimmings, which sounded like a very difficult proposition to me. However, I tried out essentially what he has described and found it quite possible to make satisfactory manganese bronze, judging the results by tensile tests, etc. The furnace used was an oil-fired revolving furnace of 1000 lb. capacity. Unfortunately, the company for which I was working did not require the bronze at that time; but my experience, small as it was, leads me firmly to believe that what Mr. McKinney has said is feasible, and I would strongly advise anyone interested to try it out.

G. H. CLAMER,† Philadelphia, Pa.—I have been more or less familiar with the work Mr. McKinney has been doing in the Washington Navy Yard, but I did not appreciate that he was carrying the consumption of scrap quite to the extent he has described.

The original manganese bronze, known as the Parsons manganese bronze, was introduced into America 25 or 30 years ago by the Wm. Cramp Ship and Engine Building Co. It was always considered an alloy of the highest order; that is, it required knowledge, experience, and the use of raw materials of the highest quality to produce it, in order to get satisfactory results. Lake copper was used and the Bertha, or Horsehead spelter. I think possibly that I was the first one to make manganese bronze in this country, independent of the Cramp Co. There was practically no information on the subject at the time I started; there was nothing in the literature, except that we knew that Parsons manganese

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\* Manager, Testing Dept., James Robertson Co., Ltd.

† First Vice-president and Secretary, Ajax Metal Co.

bronze was a very good metal and was very largely used by the United States Government. The Cowles Co., of Lockport, then introduced aluminum bronze, which carried about 10 per cent. of aluminum and 90 per cent. copper; there was a great deal of discussion and controversy, as to the relative merits of these two alloys. Finally the manganese bronze won out, because it was more easily handled and could be more cheaply produced. The Cowles Co. then undertook to manufacture manganese bronze. They manufactured the alloy by the use of manganese copper and actually introduced a considerable proportion of manganese into the alloy, and I will say that the bronzes they made at that time, carrying about 4 per cent. of manganese and as high as  $1\frac{1}{4}$  per cent. of aluminum, were as good bronzes as I have seen. These alloys had a very high yield point, good elongation, and tensile strength, in some cases as high as 90,000 pounds.

After we began to manufacture manganese bronze, several others began its manufacture, so, naturally, the producer started to look for cheaper methods of production. The first step was to use a lower grade of spelter; consequently, the intermediate grades of spelter, carrying 0.2 per cent. of lead were used. After that, there was some scrap used but the scrap was always of a strictly known quality and of low lead content. We also used zinc dross in making this alloy about 10 years ago, appreciating the fact that in zinc dross we had a ready means for introducing iron into the alloy. The alloys were made merely by the use of zinc dross, aluminum, and copper and gave very satisfactory results; these alloys contained no manganese whatever. We were very careful, however, to use only the zinc dross that came from high-grade spelter, the spelter of very low lead content, such as is produced in galvanizing wire. We found that by using drosses of that kind we could get very satisfactory results, but it was hard to obtain drosses that ran sufficiently uniform to use without a great deal of care. Some of the slabs of dross we obtained evidently were mixed with dross from the use of lower grade spelter and contained 2 to 3 per cent. of lead. Other slabs contained no lead at all, so, for that reason, the use of zinc dross was given up.

We have been led to believe that the reverberatory furnace is an exceedingly bad type in which to make manganese bronze. In this furnace it is rather hard to control the atmosphere and one of the chief difficulties that I would see in making manganese bronze as Mr. McKinney describes would be in the oxidation of the aluminum. Aluminum forms an infusible oxide, unreducible by carbon at the prevailing temperatures, which is exceedingly hard to get out of the metal. It is probably fluxed by the salt that he uses and carried to the top, but I would be a little afraid of using bronze of that kind on large and important castings.

In making the test bars, as they are ordinarily made with a coupon or in the ingot, as Mr. Jones has said, it is easy to get a good test; but when you dig right into the body of the casting, I am afraid that the results obtained by that method would be somewhat uncertain. Mr. McKinney has certainly had a vast experience; he has made lots of castings and he is not putting forth here anything that he has done in a small way but I would like to ask if he has investigated the condition of the metal, in the heart of the castings, as compared with the test bars.

The U. S. Government specifications, I might say, as Mr. Corse has also pointed out, are very rigid as regards lead content and use of scrap. This paper, coming from a representative of one of our Government departments, is significant. I think the Navy Department should be willing to modify their specifications, if they will accept material of that kind made by the Navy Yard.

C. R. SPARE,\* Philadelphia, Pa.—The writer of the paper has informed me that about 1,000,000 lb. have been made in this way in the last year; certainly this is a sufficiently large quantity to determine what he is doing. Of course, I could imagine that the use of known materials is taken for granted. The Navy Yard has probably had a large amount of materials of known compositions; that is where it had a great advantage, because the average small foundry man who will go out and purchase scrap materials of unknown composition and mixed badly certainly can not get such uniform results. Of course, the author melts his metal twice. He first applies intense heat, after which the metal is analyzed in order to determine composition, and then, after his composition is known, he remelts it. This, if done carefully, would undoubtedly give him a uniform composition of the bronze. We have not found that large percentages of manganese result in a weakened alloy; we have used manganese up to 5 per cent. without losing any dross or oxides in our castings. That is European practice also. In France and England (and I have had occasion to examine some samples from Germany and Austria) these bronzes are frequently high in manganese. In fact, some very difficult castings in submarine and torpedo-boat destroyers and air compressors ran high in manganese. Castings that had to carry an air pressure of 3 lb. per in. carried high manganese, so that I am inclined to differ with the statement that high manganese is inconsistent with clean, sound castings.

P. E. MCKINNEY.—With reference to the question of tests, I might say that you have seen the specimens passed around the room which are cut from rather heavy castings cast in sand, not against a chill. It represents a dry sand casting, a most difficult casting on which to get high physical values, on account of the extremely slow cooling.

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\* American Manganese Bronze Co.

With reference to taking tests out of the body of the casting, I feel that, so long as the shrinkages are right on the casting proper, a cut taken anywhere in a casting will be representative of the metal in an attached coupon not covered on a chill. That will hold in aluminum bronze, manganese bronze, steel, or any other metal that has a high shrinkage. We are frequently called upon to take tests for information from the casting proper, in which case I do not hesitate to remove a test piece from the castings by the hollow mill. As a matter of fact, I would just as soon accept the test piece obtained with a hollow mill as I would that from an attached coupon.

With reference to size, I have made quite a number of castings weighing up to 9.5 tons with this metal; I do not know whether there are any heavier ones. These castings, moreover, are in service and have stood rather hard service during the war in torpedo practice.

The objections to manganese and aluminum as ingredients, of course, might depend on whether you use new metal or scrap, because usually the commercial grades of manganese bronze have added to them quite a percentage of aluminum and manganese. But our experience has shown that, for a casting alloy, aluminum and manganese are very beneficial in giving toughness and strength, without necessitating an increase of the zinc content.

WILLIAM CAMPBELL,\* New York, N. Y.—I would just like to confirm what Mr. McKinney has said about test specimens taken from the casting itself. At the Brooklyn Navy Yard we have found that the test specimen taken from the casting gives just as good results as that cut from the coupon attached to the casting. As he says, the question of strength is a question of foundry practice. When the foundry tries to skimp on the metal and when sufficient head is not used for shrinkage the metal falls down. The test specimen shows cracks through practically its whole length; and under the microscope you will find that the crystals do not properly cohere. When the metal is poured right, at the correct temperature, etc., the test specimen from the casting will practically agree with that from the attached coupon.

With regard to composition, it seems that you can get equal strength and ductility, whether you are dealing with high or low manganese and iron, high or low aluminum, and up to a certain per cent. of tin. The same holds good for the amount of lead at least up to 0.25 per cent.; apparently lead does not have much effect on the strength as long as it is in the form of fine well distributed globules.

The conclusion we have come to is that the melting and foundry practices are the two great factors in manganese bronze and that in nearly all cases of failure it was found not to be the trouble with the com-

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position as much as that arising from included dross and from shrinkage, during the period of solidification.

J. B. F. HERRESHOFF, New York, N. Y.—Nothing has been said about antimony, which, as we all know, is bad in copper, especially in copper used for brass. The results are very bad. There is just the possibility that in the use of scrap, unless some change is made, we might find some hard lead. In that case, we might have brass of very low strength. We do know that if there is even a trace of antimony in the copper the resulting brass is not ductile.

I see no reason why cartridges should not be made from brass where scrap is used, if the scrap comes from the high grades of copper that all the firms are putting out now. Electrolytic-copper scrap, and scrap of that sort, ought to be perfectly satisfactory; and with proper production the brass ought to be sufficiently pure for cartridges.

WILLIAM HAMILTON,\* Newport News, Va.—Just one word to substantiate some of the statements made by Mr. McKinney. I have seen his work and know that he has made as fine castings as I have seen. My firm made the very same experiments as Mr. McKinney has described and found that a great deal depends on the foundry practice. Unless manganese bronze is gated at the bottom, you will not get pure metal, but will be sure to get a dirty casting. In one case, which Mr. Clamer is well acquainted with, we went through a great deal of experimenting to get good test pieces and found that the test piece on the bottom of a casting did not give any better elongation or reduction of area than one taken from the bottom of a riser. The riser was on the top of a casting weighing 15,000 lb., the riser was 3 ft., outside diameter, and the inside 14 in. When the test piece was taken from the riser, we got almost the same result as from the coupon attached to the very bottom of the casting, which was about 6 ft. from the top, with that much pressure on it. As Mr. Clamer said, there are a great many things we have to do for the Government that the Government does not do for itself; a great many things pass in its foundries that would not pass in ours. Consequently, we have to take tests from different parts of the castings.

As to the ingredients, I have tried this manganese bronze, getting the zinc from our own galvanizing plant, and after analyzing have found that I made just as good manganese bronze as Parsons could sell us. We have just as good manganese bronze out of all scrap, with the exception that the elongation is nearly always a little lower. Compared with the tensile strength, Mr. McKinney's elongation is a little lower. That is the only difference I have found. We made the bronze in

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\* General Foreman, Iron, Brass, and Steel Foundries, Newport News Shipbuilding and Dry Dock Co.

crucibles, never in a reverberatory furnace. I have made tons of it and put it into castings and propellers, and I have yet to find it to fail.

HENRY TRAPHAGEN,\* Toledo, Ohio.—Mr. McKinney's valuable paper has shown us that the material commonly looked upon as worthless junk can be made into not only usable, but really good material. His results form a remarkable demonstration of the application of the three great requisites of successful metallurgical practise; viz., patience, horse-sense and elbow grease.

You will note one significant remark: "If the material is properly melted, I can obtain excellent results." He might have added "even from junk." Careful melting coupled with common sense is the open sesame to the reputation of some of our most successful firms, and the lack of it, the reason for countless failures. It makes no difference whether the product be manganese bronze, high brass, steel or iron, the best materials, carelessly melted, spell failure, while poor material, properly handled, will often give excellent results.

It really seems to me that we spend a great deal of time spinning fine theories and gazing into microscopes, when we might well give a little attention to the man on the job, and the way he handles the furnace. Perhaps it is not scientific, probably it does not agree with our ideas of dignified discussion, but I have no hesitation in stating, and a fair share of experience has proved it, that 95 per cent. of foundry troubles are directly traceable, not to abstruse scientific causes, but to plain every day carelessness. For instance, about a year ago, a certain foundry made heat after heat that failed in reduction of area. Numerous experts were consulted and the opinions given would have filled a book, but still the heats continued to fail. Microscopic examinations and chemical analyses were made almost without number and without a solution of the problem. It was then decided to use horse-sense and make the men use elbow grease. The same raw materials were melted, but the metal was held under a protective slag until quiet; it was then tapped into the ladle and held for 10 min. before pouring. The molds were carefully cleaned and kept clean, and a large riser was placed over the test bar. From that day and until the end of the war, that firm did not have a single failure. Yet the same raw materials, the same furnace, the same pattern, the same annealing methods were used; in fact, nothing was changed except the method of handling.

Pig metal and scrap are always dirty and more dirt is gathered from the furnace and from the molds, and unless the metal is allowed to remain quiet and clear itself, failure is almost certain. Mr. McKinney has shown the way. Refine hot, pour cool into clean molds, after holding the metal for a reasonable time, and many of our knotty problems will disappear.

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\* Metallurgist and Chemist, Toledo Steel Castings Co.



## Non-metallic Inclusions in Bronze and Brass\*

BY G. F. COMSTOCK,† NIAGARA FALLS, N. Y.

(Milwaukee Meeting, October, 1918)

IN the literature of metallography there is a large amount of material describing the various non-metallic inclusions found in iron and steel, and the appearance of sulfides, silicates, oxides, or alumina in steel under the microscope is fairly well known. The inclusions found in non-ferrous metals are, however, not so well known, at least judging from published writings, and the author has been interested in examining small castings of copper, brass, or bronze with certain inclusions purposely mixed with the metal, to see what was the characteristic appearance of each kind of inclusion, and if they could be readily distinguished from one another.

The method employed in most cases consisted in overheating and oxidizing a small charge of copper, then cooling it somewhat, and adding a suitable amount of the element whose oxide it was desired to observe. This element was stirred in well and given time to react with the oxygen in the copper, then the melt was poured in a sand mold in the form of a cylinder about  $2\frac{1}{2}$  in. (63 mm.) in each dimension, with a sprue about  $1\frac{1}{2}$  in. (38 mm.) in diameter and 3 in. (76 mm.) high on top of the cylinder, the sprue forming the only riser. Samples for examination were cut sometimes from the center of the casting, and sometimes from the sprue or riser. A few of these samples were cast in chill molds. The appearance of sulfides was studied in an alloy containing 10 per cent. each of tin and lead, with which a flux of plaster of Paris was used. The appearance of foundry sand was studied in several alloys, one of them having sand stirred into the metal intentionally, and others having cut into the molds badly when poured, so that the castings were spoiled by included sand.

As all this work was done on alloys having copper as the chief ingredient, the natural starting point was copper oxide, the appearance of which in a micro-section is well known. Cuprous oxide is soluble in molten copper, but separates out in freezing and forms a eutectic containing 3.5 per cent.  $\text{Cu}_2\text{O}$ , or 0.39 per cent. oxygen. If there is less

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\* This paper was first printed in the *Journal* of the American Institute of Metals, March, 1918.

† Metallographist, The Titanium Alloy Mfg. Co.

oxygen than 0.39 per cent., primary crystals of copper are embedded in the eutectic; and if more than 0.39 per cent. oxygen is present, the eutectic surrounds primary crystals of  $\text{Cu}_2\text{O}$ . Fig. 1 illustrates the former condition and Fig. 2 the latter, both showing unetched samples. The oxide particles are light blue when examined by white light and contrast strongly with the red background of copper.

Copper absorbs oxygen very readily when melted, but when other elements are alloyed with it, the copper oxide is always reduced and the added element is oxidized. Tin is one of the most common additions to copper, being used in all true bronze, and its oxide takes the form of angular crystals or needles of a very dark bluish gray color, as shown in Figs. 3, 4, and 5. The first two show views of the same casting poured in sand, but Fig. 5 is a view of a chill-casting, in which the tin-oxide crystals are much smaller. Special care was taken to avoid contamination of either of these heats by anything except copper, tin, and oxygen, and in the other cases to be described later equal care was taken to keep the heats free from any undesired element. The poor polishing qualities of these tin-oxide crystals are noteworthy, as they always appear more or less pitted and standing out in relief above the metallic surface with heavy black outlines. This probably indicates that they are hard and brittle. After etching with ammonia and hydrogen peroxide, as in Fig. 4, they appeared still more pitted, as if this reagent had attacked them or partly loosened them from the metallic surface.

It has been suggested that tin oxide occurs as films surrounding the primary crystals of the bronze; and although some of these films are seen in Fig. 4, the writer believes that they were caused by gas dissolved in the metal. The reasons for this view are: the angular crystalline form is characteristic of inclusions known to be tin oxide; the inclusions are idiomorphic and scattered through the bronze crystals rather than between them; the chilled sample (Fig. 5) did not show any of the films; and similar films were found in samples containing no tin, but zinc and phosphorus, the oxides of which are volatile. The common oxide films found so frequently in commercial bronzes are therefore thought to be caused either by the volatile oxide of zinc or phosphorus or by dissolved gases thrown out of solution when the metal solidifies. These films are illustrated in Figs. 6 and 7, the latter being an extreme case, where the unsoundness was very evident before the specimen was etched. The more usual oxide films, as shown in Fig. 6, do not appear distinctly until after etching, as the polishing seems to make the metal flow over and hide them.

Several unsuccessful attempts were made to examine inclusions of zinc oxide in brass, but, at first, the only result obtained was an apparently clean though unsound metal with films and cavities like those shown in Figs. 6 and 7. Finally, some typical inclusions were obtained

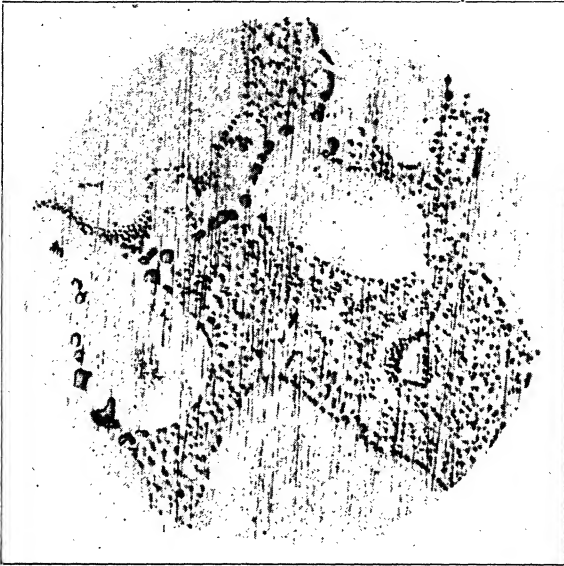


FIG. 1.—ELECTROLYTIC COPPER INGOT, SHOWING OXIDE EUTECTIC, UNETCHED.  $\times 400$ .



FIG. 2.—BADLY OXIDIZED COPPER, SHOWING OXIDE CRYSTALS AND EUTECTIC, UNETCHED.  $\times 200$ .

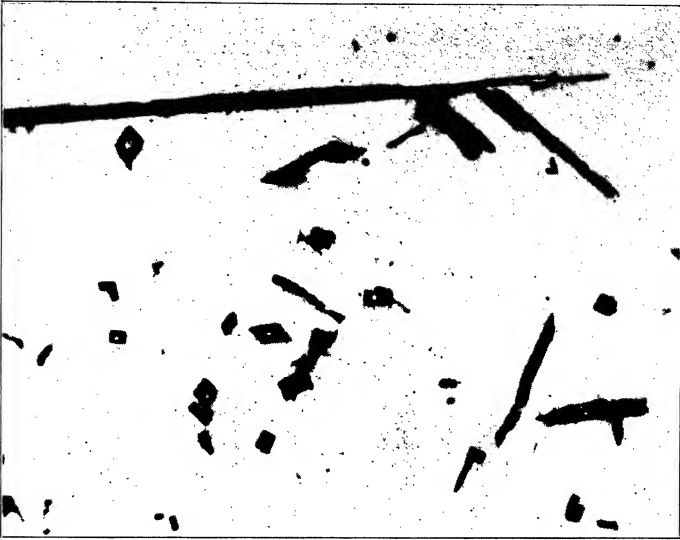


FIG. 3.—TIN-OXIDE CRYSTALS IN OXIDIZED COPPER TREATED WITH 10 PER CENT. TIN, UNETCHED.  $\times 200$ .

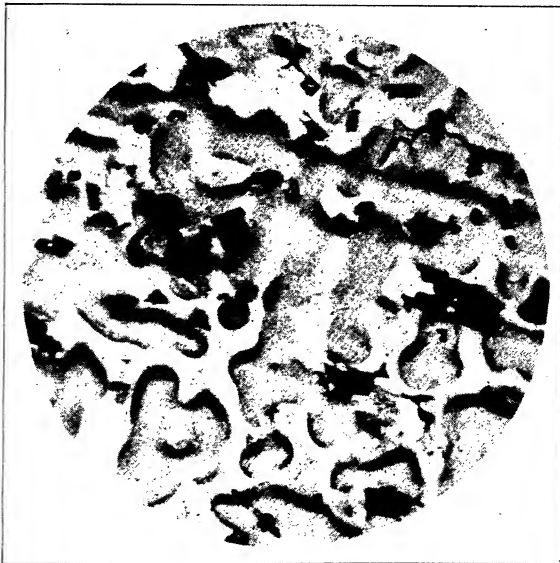


FIG. 4.—SAME SPECIMEN AS FIG. 3, AFTER ETCHING WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 100$ .

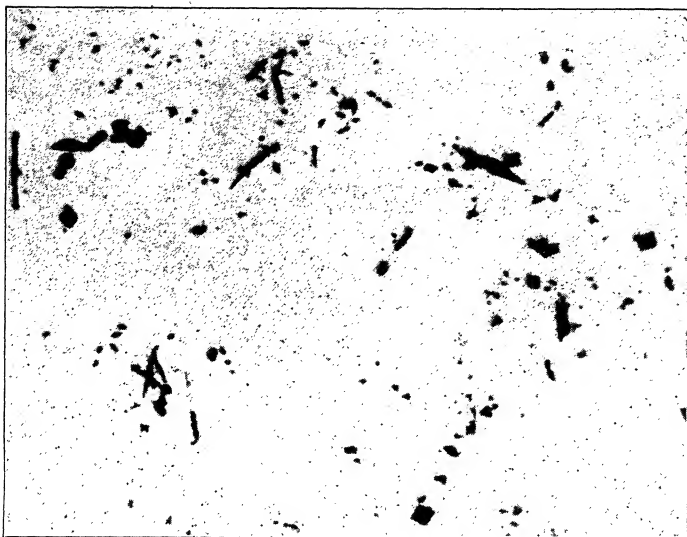


FIG. 5.—TIN-OXIDE CRYSTALS IN ANOTHER HEAT OF BRONZE MADE AS IN FIG. 3 BUT CAST IN A CHILL INSTEAD OF SAND, UNETCHED.  $\times 400$ .



FIG. 6.—TYPICAL OXIDE FILMS AND SPOTS IN GUN-METAL BRONZE CASTING, ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 400$ .

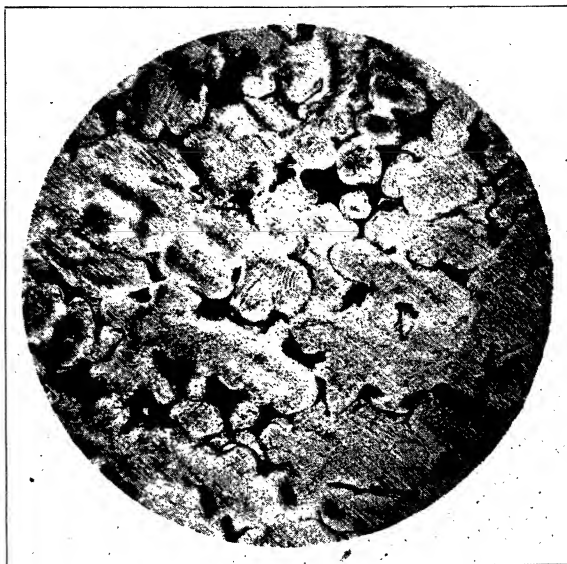


FIG. 7.—VERY UNSOUND SPOT IN BRONZE CASTING WITH 12 PER CENT. TIN AND 6 PER CENT. ZINC, ETCHED LIKE FIG. 6.  $\times 100$ .

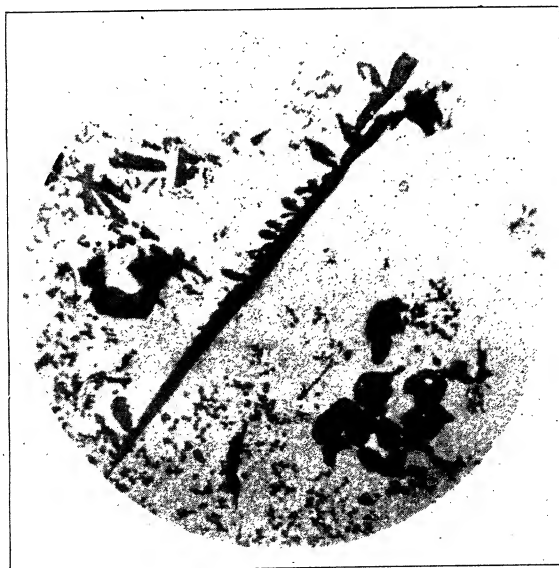


FIG. 8.—ZINC-OXIDE INCLUSIONS IN OXIDIZED COPPER TREATED WITH 30 PER CENT. ZINC, UNETCHED.  $\times 200$ .

in three cases, as shown in Figs. 8, 9, and 10. Some of these appear idiomorphic, but they are distinguished from tin-oxide crystals by their lighter color, smoother surface, without very dark outlines, and more rounded form. Fig. 10 shows metal cast in a chill, hence the inclusions are much smaller in it than in the others, which were cast in sand.

Manganese and phosphorus, when added to oxidized copper, gave results like those first obtained with zinc; that is, no typical inclusions were found although the castings were not sound. Fig. 11 shows the intercrystalline films produced when 0.5 per cent. of phosphorus was used to deoxidize some normal copper. These cavities seem to follow along the lines where the copper-oxide eutectic would be expected, had the metal not been deoxidized. Films that have been described as tin oxide are exactly similar to these, which were caused by the volatile oxide of phosphorus. Fig. 12 shows some badly oxidized copper after treatment with 2 per cent. of phosphorus. The phosphide eutectic was abundant here, but no non-metallic inclusions were found, the black spots shown being merely cavities.

While on the subject of phosphorus, it might be of interest to describe the writer's experience in identifying the phosphide in bronzes, although of course this cannot be considered a non-metallic inclusion. In specimens containing copper-tin eutectoid (or delta) and phosphide, these two hard bluish substances were found to be indistinguishable when etched with ammonia and hydrogen peroxide, or with ferric chloride. But before etching, the phosphide could be distinguished from the eutectoid because it was darker in color and stood out in relief above the rest of the alloy on account of its greater hardness. Also after etching with the above-mentioned reagents, if 50 per cent. nitric acid was applied the eutectoid was slightly attacked leaving the phosphide perfectly bright. As little as about 0.1 per cent. phosphorus can be detected in bronzes in this way. Figs. 13, 14, and 15 illustrate the appearance of phosphide in the presence of eutectoid when the same specimen is treated in different ways.

These three photomicrographs illustrate the appearance of sulfide as well as phosphide in bronze, for the alloy that they represent was made with a flux of plaster of Paris covering it in the crucible. The analysis of the alloy showed 79.3 per cent. copper, 9.9 per cent. tin, 9.8 per cent. lead, 0.4 per cent. phosphorus, 0.36 per cent. sulfur. In the unetched section, the sulfide looked rather dark blue, but when the bronze was made dark brown by the etching reagents, the sulfides appeared very light by comparison, as none of the ordinary reagents attacked them. They had more regular rounded shapes than the phosphides, which had the typical form of a eutectic, as illustrated in Fig. 15. These two substances can also be readily distinguished by the darker color of the sulfide either in an unetched section or after etching with ammonia and



FIG. 9.—ANOTHER VIEW OF ZINC-OXIDE INCLUSIONS, LIKE FIG. 8.

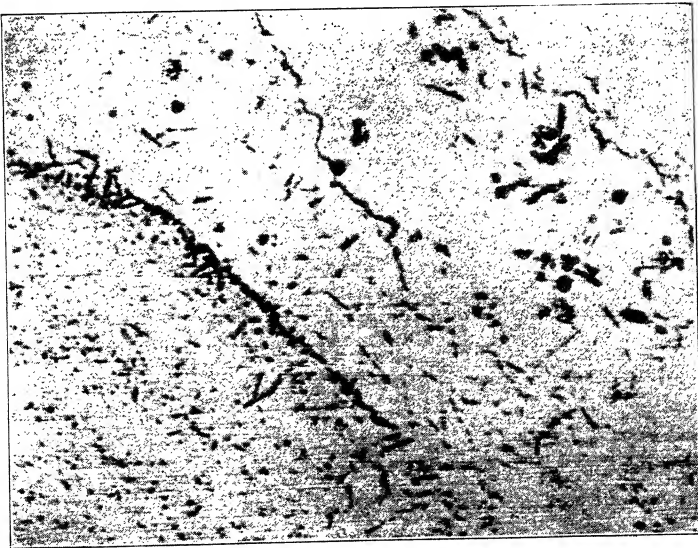


FIG. 10.—ZINC-OXIDE INCLUSIONS IN ANOTHER HEAT OF BRASS MADE AS IN FIG. 8, BUT CAST IN A CHILL INSTEAD OF SAND, UNETCHED.  $\times 400$ .



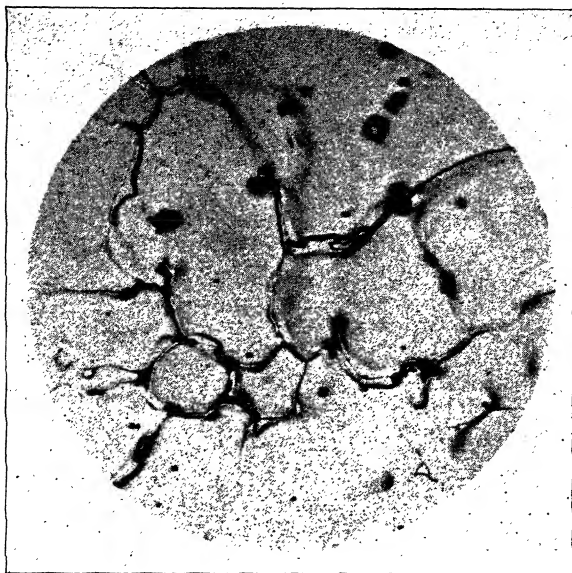


FIG. 11.—OXIDIZED COPPER TREATED WITH 0.5 PER CENT. PHOSPHORUS ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE, THEN REPOLISHED.  $\times 100$ .

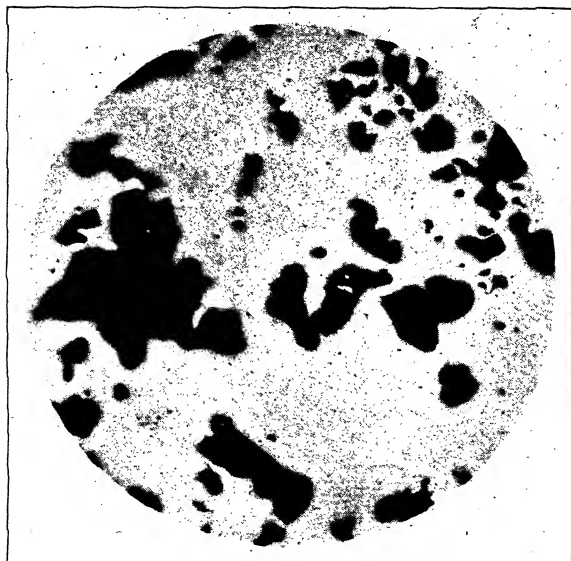


FIG. 12.—BADLY OXIDIZED COPPER TREATED WITH 2 PER CENT. PHOSPHORUS, UNETCHED.  $\times 20$ .

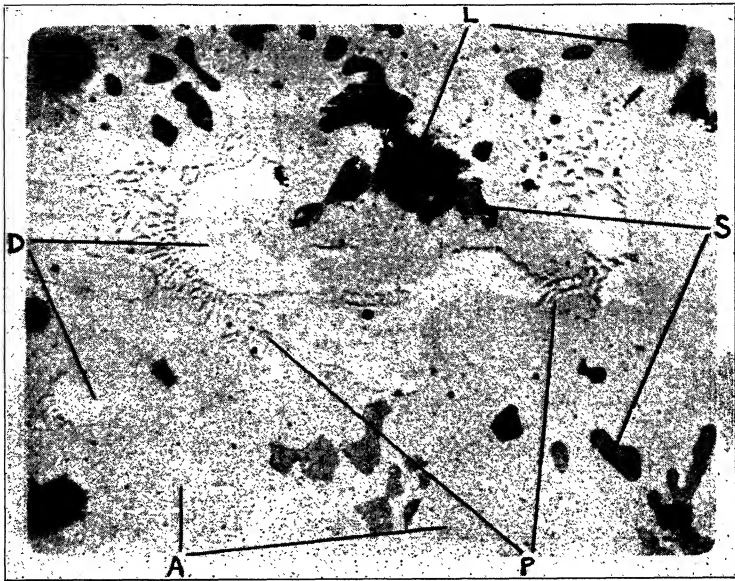


FIG. 13.—PHOSPHOR-BRONZE SHOWING BLACK SPOTS OF LEAD *L*, DARK GRAY SULFIDE *S*, BRIGHT DELTA *D*, AND LIGHT GRAY PHOSPHIDE *P*, IN FINE PARTICLES STANDING OUT IN RELIEF ABOVE THE DELTA *D*, AND ALPHA *A*. UNETCHED.  $\times 400$ .

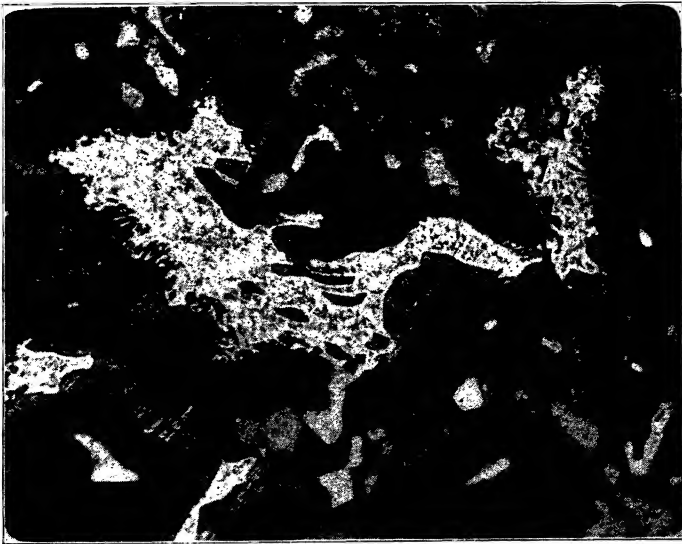


FIG. 14.—SAME SPOT AS FIG. 13, TAKEN AT SAME MAGNIFICATION AFTER ETCHING WITH AMMONIA AND HYDROGEN PEROXIDE, FOLLOWED BY FERRIC CHLORIDE. THE SOFT CORES OF THE ALPHA CRYSTALS ARE HERE SHOWN BLACK LIKE THE LEAD, AND THE PHOSPHIDE CANNOT BE DISTINGUISHED FROM THE DELTA OF THE EUTECTOID.

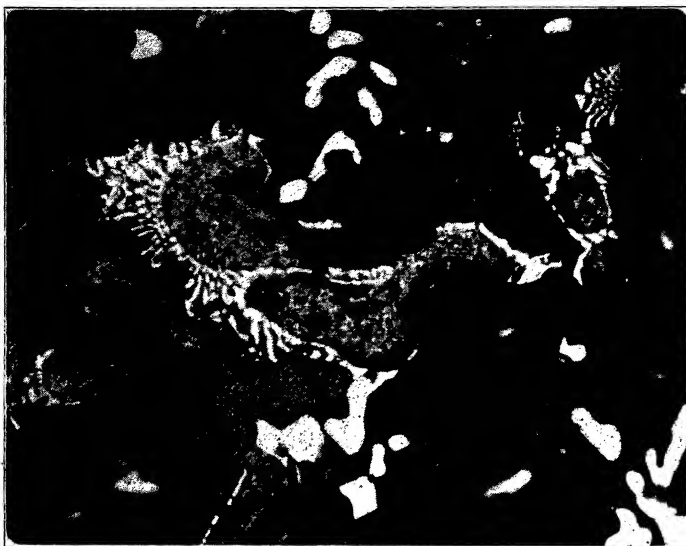


FIG. 15.—LIKE FIG. 14, BUT AFTER FURTHER ETCHING WITH STRONG NITRIC ACID. THE EUTECTOID IS HERE DISTINCTLY DARKER THAN THE PHOSPHIDE, AND THE SULFIDES APPEAR LIGHTER BY CONTRAST WITH THE DARKENED ALPHA.

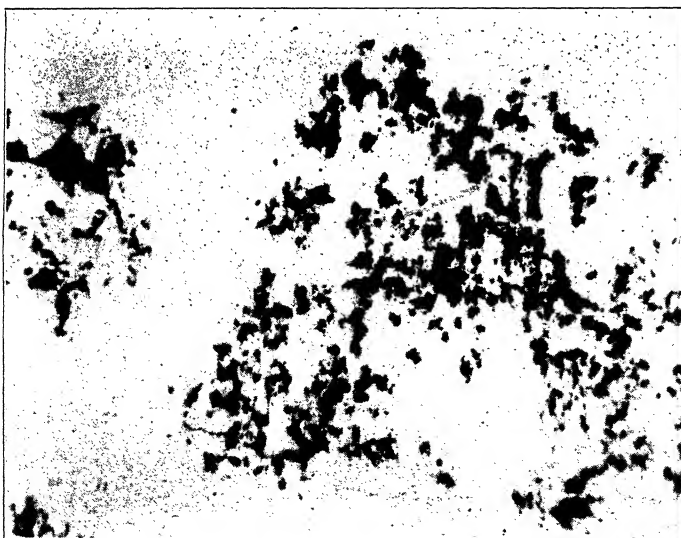


FIG. 16.—ALUMINA INCLUSIONS IN OXIDIZED COPPER TREATED WITH 8 PER CENT. ALUMINUM, UNETCHED.  $\times 200$ .

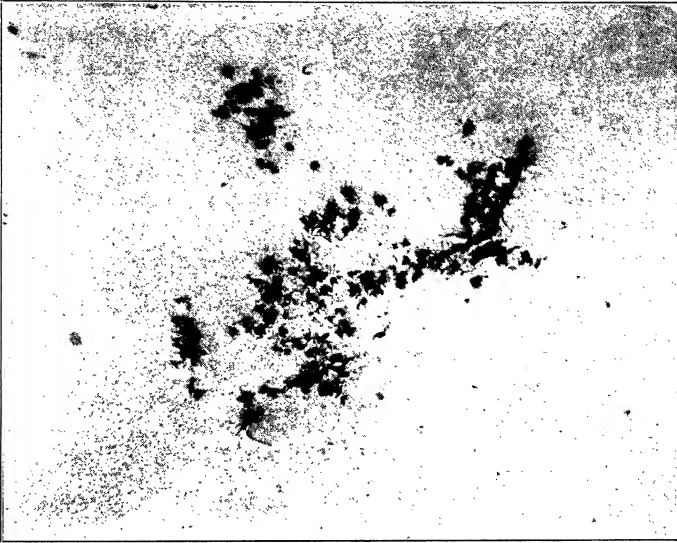


FIG. 17.—ALUMINA INCLUSIONS IN A CASTING OF 10.7 PER CENT. ALUMINUM BRONZE UNETCHED.  $\times 200$ .

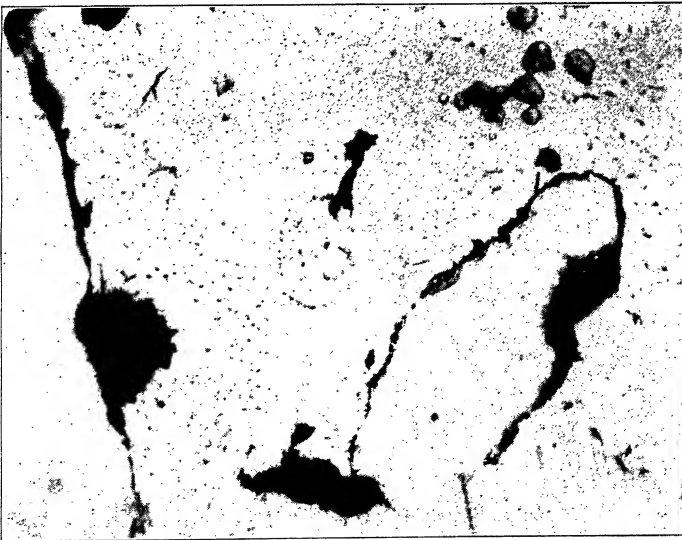


FIG. 18.—SILICATE INCLUSIONS AND STREAKS ALONG BOUNDARY BETWEEN COPPER CONTAINING OXIDE SPOTS AND COPPER DEOXIDIZED BY ABOUT 4 PER CENT. SILICON, UNETCHED.  $\times 200$ .

hydrogen peroxide. Sulfide inclusions in bronze look very similar to the few samples of zinc oxide in brass that have come to the writer's attention, but the latter were readily attacked by strong nitric acid, while no reagent has been found that darkens the sulfides in bronze.

The oxide of aluminum, alumina, has exactly the same appearance in bronze as in steel, and Figs. 16 and 17 show typical examples, the former having been made to contain alumina intentionally and the latter showing these inclusions in a regular aluminum bronze casting. The black specks shown are probably pits in the polished surface, where particles of alumina have fallen out, as these particles themselves are rarely seen in a



FIG. 19.—MASS OF SILICATE SLAG FORMED BY REMELTING OXIDIZED COPPER AND SILICON COPPER TOGETHER, UNETCHED.  $\times 200$ .

polished section. The very dark color, small size, close grouping yet lack of coalescence, irregular shape, and rough edges due to pitting are characteristic of alumina in bronze or cast steel.

It was thought that silica might have the same appearance in bronze as alumina, but the writer has been unable to secure any evidence in support of this view. Several attempts to produce silica in copper by oxidizing the metal and adding silicon were not very successful, and no typical silica inclusions were found. Fig. 18 shows one of the results from these attempts. Part of the view shows oxidized copper, with pale round spots of  $\text{Cu}_2\text{O}$ , and part shows silicon copper, clean and sound. Between the two are streaks and spots of a very dark material, most of which has apparently fallen out of the section in polishing. This may be

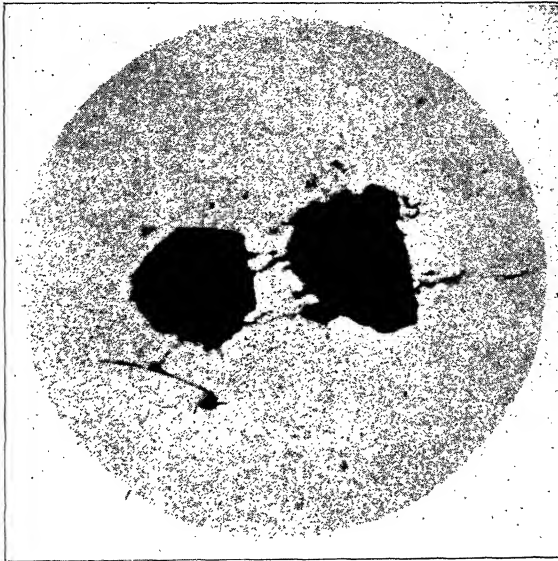


FIG. 20.—TWO SMALL GRAINS OF SAND IN ALUMINUM BRONZE, UNETCHED.  $\times 200$ .

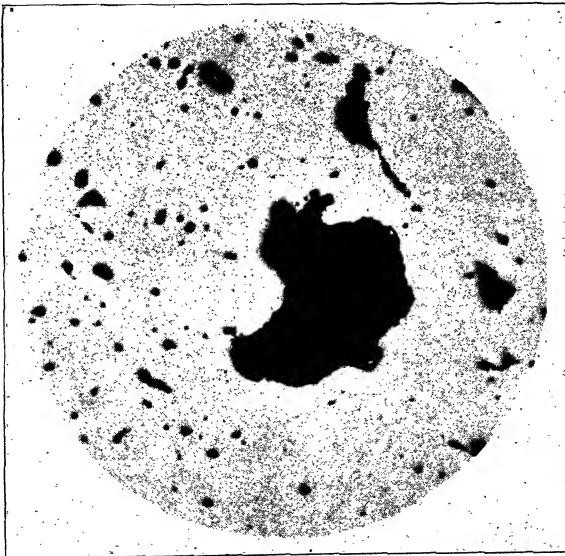


FIG. 21.—A LARGER GRAIN OF SAND IN A BRONZE CASTING WITH 9 PER CENT. LEAD, UNETCHED.  $\times 50$ .

silica or a silicate. It has no resemblance to alumina, but resembles slag in steel quite closely. Fig. 19 shows a mass of silicate or slag included in a chill casting made of oxidized copper and silicon remelted together several times. Some copper oxide is still present in this specimen, but most of the material had been deoxidized by the silicon, and the oxidized product or slag had coalesced into fairly large masses and left the silicon copper clean. The slag here is not homogeneous, but seems to contain particles of copper oxide or some similar substance.

Figs. 20 and 21 show specimens of foundry sand included in bronze castings, the former being aluminum bronze with sand stirred in intentionally and the latter an ordinary leaded bronze that had burned into the mold excessively and so become mixed with sand at the surface. The angular form of the inclusions in Fig. 20 would indicate that they were grains of sand and not slag formed in the metal, but this distinction is not always so clear. In general, it is probably reasonably safe to say that where smooth dark-gray inclusions like these are angular in shape or have convex sides, they are sand grains; and when their sides are concave, they are slag inclusions. The writer is not aware of any other distinction between slag inclusions and grains of sand either in bronze or cast steel. It should be noted in this connection that it is more difficult to polish a sample of bronze containing sand than a similar sample of steel, without having the sand fall out leaving merely a pit or cavity in the finished section. The cavities thus left cannot generally be distinguished from cavities caused by shrinkage, gas, or a volatile or powdery oxide of some element like phosphorus or zinc. Another point that might be noted in regard to sand inclusions is that when a casting containing sand beneath the surface is machined, the tool naturally pulverizes the sand, and the white powder thus produced may readily be mistaken for an oxide in the metal, such as alumina. The particles of alumina in bronze or steel, however, are so fine and so intimately mixed with metal, as shown by the microscope, that in machining, while the point of the tool may be dulled by them, it is very doubtful if they ever break out of the metal chips in sufficient quantity to be recognized as a white powder.

In presenting these notes the writer does not claim to have made any remarkable discovery or to have found the best method for identifying the non-metallic inclusions in non-ferrous metals, nor does he claim to be able to identify every inclusion that may occur in a bronze. It is hoped, however, that the notes may be of some interest or possible value to other metallographists, and may bring out criticisms or further suggestions along these lines which will accelerate progress toward the true interpretation of everything that we see in metals with the microscope.

## Standards for Brass and Bronze Foundries and Metal-finishing Processes\*

BY LILLIAN ERSKINE,† NEWARK, N. J.

(New York Meeting, February, 1919)

WHILE brass and other copper alloys have long been listed as offering health hazards to their workers, it is questionable if the metals involved are alone responsible for the trades' records of morbidity. The entire science of foundry sanitation is the product of the past two decades; and the best standards now operative would have been regarded as little short of revolutionary prior to 1910.

The special handicap to the conservation of the health of the brass founder's employees has been the fact that, unlike the iron founder, his business could be profitably carried on in almost any small and unsanitary quarters. As a result, writers on occupational disease have accepted brass chills or brass founders' ague as an inevitable feature of the trade; and respiratory disabilities, as well as phthisis and disturbances of the nervous system, are also included as prevalent among those employed.

While there can be no question as to the basic accuracy of past investigations, there is every reason to doubt the necessity for an undue percentage of ill health among brass molders and casters who are safeguarded by scientific ventilating standards and by improving personal habits of sobriety. There is, however, need for a campaign of education as to the most approved practices and equipment whereby the employer may safeguard the health of his workers, and thereby reduce his labor turnover and increase his productive efficiency.

Broadly speaking, copper, tin, and aluminum carry negligible health hazards. The toxic qualities of lead, however, are a menace; especially if scrap lead is recovered or bought for foundry processes. The rapid volatilization of zinc renders its fumes and flocculent condensation the chief problems in conserving the health of the brass worker. The frequent presence in the foundry air of traces of arsenic compounds, lead

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\*Prepared for Lewis T. Bryant, Commissioner of Labor of the State of New Jersey.

† Investigator of Occupational Diseases for the New Jersey Bureau of Hygiene and Sanitation; John Roach, Chief.



oxides, carbon monoxide, oil fumes, and occasional mineral acid and alkali vapors, from cleansing and dipping processes, emphasizes the need for free natural or mechanical ventilation.

On the storage and transportation of the metals needed in furnace processes depends but to a minor extent, the amount of dust in the air. Where scrap lead is used, the poisonous dust accumulating in bins, or scattered during transportation, constitutes a hazard.

The use of stick phosphorus, instead of a phosphor-copper or phosphor-tin, as a scavenger in bronze is now practised in the small foundry only for economic reasons. Special instruction and warning as to its highly poisonous character and combustion-hazards are necessary in the case of new employees.

### FOUNDRY CONSTRUCTION.

In foundry construction light and ventilation are the prime requisites. The English requirement of 2500 cu. ft. of air per brass worker engaged in sand foundry casting and of 3500 cu. ft. per worker in rolling-mill casting (or similar shops where continuous pouring is practised in foundries devoid of mechanical ventilation) is a reasonable standard for structural dimensions.

The mere provision of air space, however, is no guarantee of satisfactory productive and health standards. Adequate lighting calls for the provision of window and skylight area on a basis of 40 per cent. of the floor area, provided these are properly located and cleaned at least monthly. Fume and dust generally decrease the average of natural foundry illumination at the rate of 0.0625 to 0.25 per cent. daily. While the height of the side walls should be governed by the size of the foundry, a minimum of 15 ft. is essential for free natural ventilation.

Batteries of pivot center-hung windows satisfactorily meet the requirements of both ventilation and light. Illumination may be intensified by the substitution of wire glass for sections of the usual tight-construction roof of the monitor or side bays. Flat wire-glass skylights (hinged at the upper end to permit of their partial raising) will provide additional light and ventilation. The "hot-house" glass type of foundry construction, however, involves excessive sun exposure during 8 mo. of the year. Sawtooth roof construction gives admirable lighting but does not ensure satisfactory removal of fume and heat.

Since a southern exposure offers excess sunlight on the molding floor, it is well to install offices, core rooms, etc. on the south side of the building, leaving the west, north, and east walls free for lighting and ventilation of founding processes.

In sand-casting operations, the ingress of fresh air must be provided by windows; preferably pivot center-hung and operated in batteries. As too strong a draft of air may develop spills, during the cold months all

doors should be vestibuled except in the melting department and there also should be a convenient method of floor control over window openings.

In continuous pouring processes there should be a liberal provision of side doors, as well as horizontally or vertically pivoted windows. There should be an upward sweep of air from or near the level of the floor; no disadvantage to the product from chilling need be feared.

### ROOF VENTILATION

While the new type of inverted center peak roof gives exceptionally satisfactory results, the monitor type is still universally accepted as the standard. The angle of the roof peak should not fall far below  $30^\circ$ , as the upward rush of fume is easily checked and deflected by flattening the apex. The width of a monitor should be at least 15 per cent. of that of the building and 20 per cent. is better as a wider monitor gives better lighting facilities. In a monitor having a greater width than 20 per cent. there is a probability of the chilling and backing of zinc fumes. The height of the side walls of the monitor should never fall below 4 ft. when open slat louvers run its entire length.

Pivot center-hung windows, when installed, should run the length of the monitor and be at least 3 ft. in height, operated from the floor, and so swung through an arc of  $90^\circ$  that they may be kept in a horizontal position in clear weather, thereby creating a continuous draft through the monitor and preventing a downward current of chilled air.

Ordinarily, the monitor runs the full length of the building; but a type of cross-monitor construction, above cross batteries of pit furnaces and flasks, has been successful in preventing zinc fumes from being driven in the faces of casters to leeward of the prevailing air currents.

However satisfactory a liberal provision of the cowl or siphon type of natural-draft ventilator may have proved for general foundry conditions, the evolution of zinc fume in the pouring of yellow brass requires a freer roof ventilation than can be provided by any of the present models. This does not apply to individual installations over batteries of open-flame tilt furnaces, which are noted later.

### FURNACES AND HOODS

The pit crucible variety of furnace has long been the standard type for the brass and bronze industries. Open-flame oil and gas-fired furnaces, however, are now being used. The chief hygienic advantages of the pit crucible are: The initial melting fumes are almost invariably removed by the requisite chimney or stack draft; in tight-roof foundries mechanically ventilated hoods can be constructed above the batteries of fires, ensuring the safeguarding of the worker during incidental charging and skimming processes; the brass (usually yellow and carrying a high

percentage of zinc) is poured direct from the melting crucible, thus eliminating the double fume exposure called for in tilt furnace processes. The chief disadvantage of the pit furnace is the radiation of excessive heat. This may be overcome by a basement construction, which leaves at least a 6-ft. passageway (ventilated by flues or windows direct to the outer air) in front of the battery of fires. Ashes can be removed through this basement and the danger of a slow melt, from starved drafts dependent on working-floor conditions, is avoided.

### STACKS

The question of adequate draft for the pit furnace is governed by the diameter and height of the chimney and the number of fires it is designed to carry. Five fires or less call for a minimum stack diameter of 18 in.; thirty fires require a minimum diameter of 38 in.; and forty fires require a minimum of 45 in. Generally speaking, the height of the stack should be from twenty to twenty-five times the diameter; but adjacent buildings may make it necessary to increase these figures. The chilling of ascending heated currents of air by excessive height, however, is often responsible for the backing up of irritating and injurious fumes.

The chilling of ascending air currents is a greater handicap in the metal stack than the brick chimney. If preheating is necessary, it may be accomplished by introducing steam through a cock situated near the base or by burning wood to induce an up-draft before the actual fire is laid. The jacketing of an unlined metal stack by means of an outer shell, allowing about 2 in. of air space between it and the circumference, will prevent chilling and backing of fumes. A firebrick lining accomplishes the same result.

### HOODS

In old-type foundries, the installation of a ventilated metal hood over the pit fires is a safeguard against incidental fumes and excessive heat. The area of the hood should equal the area occupied by the battery of fires and the pit and its lower edge should not be more than 6 ft. 6 in. above the floor level. Straight, natural-draft stacks direct to the outer air, not less than 24 in. in diameter, and in a proportion of one to every 10 ft. of hood length, may be preferably employed. If, for structural reasons, the ventilating stack leads to the main furnace stack, elbows should be avoided and the ventilating stack should enter the furnace stack at an angle of not more than 45°. Fans and a standard pipe system are requisite when free roof ventilation cannot be secured or the location of the main stack does not permit of satisfactory connection.

Where the battery of pit fires is located against an outer wall, center-hung windows, in a proportion of one to every five fires, should be provided.

Since air currents always short-circuit along the lines of least resistance, free window ventilation or excess pull on a ventilating hood may hinder the pit-draft and a slow melt result. This may be prevented by the basement type of construction already noted; or by pit flues leading direct to the outer air. A pit 6 ft. deep by 7 ft. wide ensures freedom from the effects of short-circuiting.

### GRATINGS

Grating bars should be set in the frame and not cast, thereby avoiding warping and accident hazards. Metal plates with staggered perforations may be used; but care must be taken against starving the pit draft by too scant provision of openings, unless special flues are provided. In continuous casting shops, the old-fashioned movable oak plank (in spite of accident risk) is frequently employed. The basement corridor construction permits a tight metal working floor, as the problem of excess heat is practically eliminated and all ashes are removed from below.

### GAS AND OIL-FED FURNACES

The open-flame oil furnace always requires hooding and direct stack ventilation to the outer air. When the roof construction prohibits natural draft, a deep hood and direct fan pull, unobstructed by elbows, is essential for the removal of fume and heat. The practical separation, by means of hooding or partial partition of the melting from the pouring process, is of unquestioned advantage.

On a 60-in. open-flame oil furnace, the angled, oblong bell of the hood should be at least 10 ft. long by 5 ft. 6 in. wide and installed at a height of not more than 6 ft. 6 in. above floor level. On a 42-in. furnace, the hood should be at least 6 ft. 6 in. long by 40 in. wide. When ordinary sheet iron is employed, the limited area upon which the direct flame impinges should be reinforced by an inner skin preferably of  $\frac{3}{16}$ -in. steel, which can be easily renewed, thereby conserving the life of the hood.

The single stack on the hood of a 42-in. open-flame oil furnace should be at least 30 in. in diameter; for the hood of a 60-in. open-flame oil furnace, the 36-in. stack is required. Where a double stack is installed over a 60-in. furnace, each stack should be at least 22 in. in diameter. A height, over all, of from six to eight times the diameter of the stack is sufficient, unless adjoining buildings make an increased height necessary. Since the danger points from brass fumes in skimming and initial pouring are located within the area protected by the hood, its size and ventilating capacity are of importance.

The same standards apply to tilt furnaces in the bronze foundry; where the less obvious hazard of arsenic fumes and products of combustion may prove a health menace.

Where a battery of Rockwell furnaces is located against an outer wall, a hood or metal curtain should be dropped from the roof over and in front of the line to within 6 ft. 6 in. of the working platform. Center-hung windows, in a proportion of one to each furnace, should be provided in addition to natural draft or revolving ventilating hoods direct to the outer air. If the wall can only be opened up near the eaves, wall fans may be installed. Where a dead wall exists but roof ventilation is possible, the hooded area should be provided with revolving ventilating hoods of an approved type, direct to the outer air. These should be in a proportion of one to every two furnaces and have a minimum diameter of 48 in. Where natural-draft stacks are employed, the same diameter is needed and a minimum height over all of six to eight times the diameter.

Where a battery of furnaces is located in or near the center of the workroom, a hood at least 10 ft. wide by 8 ft. high should be installed above the entire line, and dropped to within 6 ft. 6 in. of the floor. The ventilating provisions should be identical with those just described.

#### VENTILATING SYSTEM

For satisfactory mechanical ventilation of the tight-roof foundry, the cubic contents of the space from which air must be removed, as well as the frequency of change called for, should be scientifically computed. By installing a well-designed pipe system, with its branched openings dropped to within 3 ft. of the floor and connected to a housed fan, a steady change of air every few minutes can be maintained at a low expense of upkeep. Almost any of the numerous bladed types of exhaust fans may be used; but, generally speaking, the smaller the fan unit, the greater is the total horsepower expended for the given change of air.

In handling 6000 cu. ft. of air in a typical cellar foundry 20 by 30 by 10 ft., Table 1 may be taken as a guide. This table shows the relative

TABLE 1.—*Ventilation Data for Small Cellar Foundry*

Number of Exhaust Fan	Diameter of Exhauster Inlet, Inches	Horsepower Required	Cubic Feet of Air per Minute Delivered by Exhauster
3	17¼	2.48	4040
3½	20	3.85	5750
4	22¾	2.24	5740
4½	25¾	1.71	6130
5	28½	0.98	5870
5½	31½	0.65	5800
6	34¼	0.50	5970

costliness of upkeep, in horsepower expended, for a small fan installation. Even when driven at the higher speeds, the skin friction developed in too small a pipe makes the seeming initial economy an ultimate financial

handicap. A No. 3 fan handles but 4040 cu. ft. of air per min., with 2.48 horsepower, through a pipe having an area of approximately 1.5 sq. ft., and at a velocity of some 2666 ft. per min. A No. 6 fan easily handles 5970 cu. ft. per min., with less than  $\frac{1}{2}$  horsepower, through a pipe having an area of about 6.2 sq. ft., with a velocity of approximately 962 ft. per min.

Table 2 may be used to determine the size of the exhauster that will most economically provide the air change that healthful working conditions demand: In the case of a typical old-type foundry 20 by

TABLE 2.—*Exhaust Fan Data*

Number of Exhaust Fan	Diameter of Exhauster Inlet, Inches	Horsepower Required	Cubic Feet of Air per Minute Delivered by Exhauster
3	17 $\frac{1}{4}$	0.13	1490
3 $\frac{1}{2}$	20	0.17	2030
4	22 $\frac{3}{4}$	0.22	2650
4 $\frac{1}{2}$	25 $\frac{3}{4}$	0.28	3360
5	28 $\frac{1}{2}$	0.35	4150
5 $\frac{1}{2}$	31 $\frac{1}{2}$	0.42	5020
6	34 $\frac{1}{4}$	0.50	5970

30 by 10 ft. containing 6000 cu. ft. of air, a No. 3 exhaust fan using but 0.13 horsepower will give an approximately complete air change every 4 min. This 4-min. change is a safe minimum standard for a tight-roof foundry, provided local skimming-hood protection is provided in addition.

The various fittings and accessories must conform to the standards of the New Jersey Bureau of Hygiene and Sanitation.

#### LOCAL SKIMMING HOODS AND WORKING PLATFORMS

Yellow-brass foundries should install local exhaust hoods for the removal of skimming fumes; these are connected to the main exhaust system and are usually dropped beside the brick or iron supports of the bays. A 14-in. pipe flaring to a 24-in. hood at a point 3 ft. above the ground, the back of the hood being dropped to the floor level leaving a projecting half moon above the crucible, gives the best results. An air velocity of 3000 lin. ft. per min. should be maintained.

The bars of working platforms of stationary furnaces should be inset, not cast in the frame. No solid metal plates should be used, because of radiated heat.

#### SPECIFICATIONS FOR EXHAUST SYSTEMS

The faulty construction of blower systems, due to lack of specific engineering standards, is the greatest handicap to satisfactory fume, heat,

and dust removal in the foundry. The questions of material and the scientific coördination of all details that will aid the flow of air generated, without increasing the cost of operation, must be fully understood to insure satisfactory results. Standard specifications are provided by the New Jersey Bureau of Hygiene and Sanitation.

### FOUNDRY HEATING

The question of equalizing extremes of temperature by means of a mechanical heating and ventilating system is of prime importance to the employee and the employer in the larger sand-casting foundry. Few industrial health hazards are more depressing to vitality or more psychologically and physically conducive to intemperance than the damp chill of the unheated foundry. The prevalence of rheumatism and respiratory diseases among molders and casters is in some measure due to lack of dressing-room and washing facilities; but the prime cause for molders is the blanket of moisture-laden air above the damp molding floor of the one-shift plant, the temperature of which is not sufficiently raised to ensure evaporation until pouring takes place, and then only to a localized degree.

The best modern heating practice relies on a forced system of warm air diffusion by means of a housed fan and pipes. The fresh-air supply is drawn from the outside over a bank of steam coils, put through the fan, and then delivered through ducts to various points within a few feet of the floor level. By this means the coldest foundry may be heated in zero weather within from  $1\frac{1}{2}$  hr. to 1 hr., the cold air in the foundry being rapidly displaced by the volume of heated outside air circulated by the fan. During preliminary heating the fan can be speeded to its maximum delivery; but as soon as a sufficient temperature has been reached, its normal speed can be resumed and maintained.

In addition to the advantage of placing the heated air at practically the floor level, this method permits thermostat control. By this means the amount of steam supplied through the heating coils can be automatically regulated; so that the temperature can be kept within  $2^{\circ}$  of the desired standard ( $55^{\circ}$ ) regardless of the outdoor temperature or the negligence or whims of any person. The necessity for guarding against the freezing of the molding sands dictates the maintenance of a safe minimum temperature at all hours during extreme weather.

### ARTIFICIAL LIGHTING

Foundry work is generally reckoned (in standard lighting codes) as requiring from 2 to 4 foot-candles. Whenever, owing to weather conditions or structural defects, natural light falls below this intensity,

artificial lighting must be used and may be roughly reckoned, with a good overhead system, on a basis that 1 foot-candle (spherical) per square foot of floor area will produce an illumination of about 3 foot-candles. A code of lighting has been prepared by the Bureau of Electrical Equipment, New Jersey Dept. of Labor.

The advantages of a good modern tungsten-lamp equipment in the foundry are: Increased production, greater accuracy in workmanship, reduced accident hazard, reduction of eye strain, reduction of labor turnover due to more cheerful surroundings, lessening of fatigue and shop friction, promotion of good housekeeping, and improved facilities for superintendence and inspection. Owing to prevailing smoke and dust exposure, the lighting equipment deteriorates at the rate of 0.0625 to 0.25 per cent. daily. As in the case of windows and skylights, a regular follow-up system of cleaning should be maintained.

### FLOORS AND GANGWAYS

The value of materials handled in the brass and bronze foundry does not permit a sand molding floor. Cement is more popular than brick with the workers, and metal-plate gangways are frequently adopted. Belgian brick makes a satisfactory molding floor. A layer of sand insulates the brick against incidental hazard from molten metal, and the sand can be easily collected and renewed when its periodical recovery-treatment for valuable content becomes necessary.

To avoid accident, it is essential to guard against excess moisture on all cement and metal-plate surfaces; and to set a minimum standard of 5 ft. for all main gangways along which the ladles must travel.

### FLASK RACKS

Where small sized standard products are habitually handled, the use of rows of fixed metal racks, or horses, upon which the flasks may be laid before pouring, conserves the physical efficiency of the molder, by the resulting saving in foot-pounds of energy ordinarily expended in stooping to the floor-level.

### PATTERN SHOP

All dust and refuse material produced by woodworking machinery in the pattern shop should be removed by an exhaust system, constructed in accordance with standard specifications, and provided with a device for injecting steam into the pipes to extinguish any blaze. Not only the conservation of health, but lessening of fire hazards dictate



such an installation. (See Standards of the New Jersey Bureau of Hygiene and Sanitation.)

### CORE OVENS

No type of oven, whether of the drawer or revolving-table variety, is always fume-tight during filling and discharging operations. Even though the oven is equipped with reasonably effective exhaust ventilation, a mechanically ventilated hood should be installed above the working doors. The hood should be of the same width as the core oven; and where only one set of doors or drawers is used, may be of the half-moon type overhanging the side where fumes are detected. A 5-in. ventilating pipe through which an air velocity of 1000 lin. ft. per min. is maintained is sufficient to care for every 5 ft. of oven width.

Where the core ovens are installed in a double-ended battery, worked on both sides, a hood should be suspended over the entire installation and mechanically ventilated by means of 5-in. pipes in the proportion of one to every 25 sq. ft. of hood. If natural-draft ventilation is relied upon, the ventilating pipe should run direct to the outer air, or enter an adjacent stack at an angle not to exceed 45°. The diameter of such a pipe should be at least 14 in. to every 25 sq. ft. of hood area.

### TUMBLING BARRELS

All barrels should be equipped with exhaust pipes of the proper proportions. A minimum diameter of 4 in. is essential, and the suction generated may vary from 1 to 2 in. (static method) according to the character of the dust.

### BUFFING, POLISHING, AND GRINDING

All dust generated in the process of buffing, polishing, and grinding should be removed by an exhaust system constructed in accordance with the details given in Table 3. A satisfactory balance must be maintained between the air exhausted and the supply admitted to take its place. Ingress openings should be at least three times the area of the egress openings. If evenly distributed, and if the air is admitted over warm steam coils in winter, a fairly draftless ventilating system can be maintained. Wheels of a special type may require larger pipe sizes than those indicated. (See Standards New Jersey Dept. of Labor). Steel safety hoods or some other approved safety device should be installed on solid grinding wheels.

All emery belts should be equipped with exhaust pipes, the size of which must be determined by the width and operation of the emery belts.

TABLE 3.—*Size of Branch Pipes for Buffing, Polishing, and Grinding Wheels*

Diameter of Wheels, Inches	Size of Branch Pipe for	
	Rag Wheels, Inches	Hard Wheels, Inches
6 or less	3.0	3
7 to 12	4.0	
7 to 16		4
13 to 16	4.5	
17 to 24	5.0	5
25 to 30	6.0	6

## PROPORTIONS OF EXHAUST FAN AND PIPING

The inlet of the exhaust fan should be at least 20 per cent. greater in area than the combined areas of the several connections to the hoods, and this increase should be carried proportionately in the main pipe throughout the entire trunk line. The piping on the outlet of the exhauster should also be at least 20 per cent. greater in area than the combined areas of the several connections to the hoods. The main trunk line should be provided with suitable cleanouts not more than 10 ft. apart, and the end of the main trunk line should be blanked off with a removable cap placed on the end.

## TEST

Sufficient suction head should be maintained in each branch pipe within 15 in. of the hood to displace 2 in. of water in a U-shaped tube. The pressure is to be taken by pressing the tube attachment over a small opening through the pipe, commonly called the static method. Tests are to be made with all branches open and unobstructed.

## SAND-BLAST ROOMS AND APPARATUS

Sand-blast cabinets should be provided with reasonably dust-tight flaps or similar protective device over the two openings through which the workers' hands must be inserted and should be mechanically exhausted by means of at least a 3-in. pipe connected to a fan system, through which a linear air velocity of at least 2000 ft. per min. is maintained. If a cover of fine wire gauze protects the back of the glass window through which the operative watches his work, the glass will not become clouded by the abrasive action of the sand. Working gauntlets should be provided and all parts of the cabinet kept in repair to guard against leaks.

On larger work and that which requires cleaning, the commercial

types of mechanically exhausted sand-blast barrels and rotary tables are both sanitary and satisfactory.

When the character of work handled is of too great a size to permit the employment of the rotary table, only the most approved make of air-tight and mechanically exhausted sand-blast room should be installed. It is essential that in addition to the overhead fresh-air supply, the floor be provided with a grating and be exhausted by means of a powerful down pull and that the operative be provided with special clothing and a helmet. In an efficient equipment the air of the room is completely changed every 6 sec.

### BABBITT AND LEAD ALLOYS

As the lead content of white-metal bearing alloys may run as high as 90 per cent., the control of the health hazards involved in their production is needed. Scientifically speaking, there is no hazard from lead fume, as the temperature of the kettles is never allowed to run high enough to cause actual volatilization of the lead. The danger of lead poisoning is from the oxides that form on the surface of the molten metal, and are scattered as impalpable dust by every draft, or by the operation of charging and skimming.

When pig lead is habitually used, the health of the worker may be protected by means of a mechanically exhausted crescent-shaped hood installed at the back of the kettle. This open-shield hood covers from  $33\frac{1}{3}$  per cent. to 50 per cent. of the rear of the kettle, leaving the working side free. It is ventilated at its base by a pipe connected to a fan, and the minimum pull of 1000 lin. ft. per min. is sufficient to induce a steady air current away from the worker.

When scrap lead is used and when old bearings are melted out, etc., a tight exhaust hood is necessary; also extra cleanliness should be observed on the part of the workers. In all lead-kettle processes, metal receptacles should be used for skimmings and no lead materials should be allowed to accumulate on the working floors.

### DIPPING

No fumes caused by metal dipping in acid or alkali solutions should be permitted to contaminate the air of the workroom. They should be promptly removed at the point of origin by means of a boxlike duct installed at the back of the crocks commonly used. This duct should be constructed of wood or metal. The main duct should have a cross-sectional area at least equal to one-half the combined areas of exhaust openings. The bottom of main duct should be 7 in. wide; the front should incline at an angle of  $115^\circ$  to the bottom. Exhaust openings with an area equal to 25 per cent. of the area of the liquid giving off the fumes

should be located as near the point of origin of the fumes as the nature of the work will permit. A velocity of air of 1000 lin. ft. per min. should be generated in each exhaust opening. If sheet metal is used in the construction of the duct, an acid-resisting paint should be used to prevent corrosion. The exhaust fan should be treated with acid-resisting paint to prevent corrosion. Floor gratings should be provided and rubber gloves for the operatives.

### PLATING

It is essential that a system of mechanical ventilation ensure freedom from stagnant air in the plating room. This may be accomplished by means of wall fans, when the tanks are located near the side of the room; or by overhead hoods mechanically exhausted; or with ventilating stacks, when the plating operations are carried on in the middle of a department. A natural-draft hood, to be effective, should only depend on a stack or stacks direct to the outer air, in a proportion of a square 12-in. stack to every 36 sq. ft. of hood area. Wooden gratings should always be provided in the plating room and rubber gloves should be provided for the operators.

### ENAMELS AND LACQUERS

The solvents for the pyroxylin, which forms the basis for the lacquers and enamels used on metal-finishing, are not only highly inflammable but poisonous to those exposed to their fumes. Wood alcohol, benzol, amylacetate, amylalcohol, volatile ketones, and acetone are (except the last two) admitted to be injurious to all who breathe them.

The fumes evolved during dipping or spraying should be controlled at their point of origin. Mechanically exhausted spray booths are now in the market, or may be "home made;" but the only type that should be chosen is one with an overhung top and a sufficient fan pull on the exhauster to ensure an air motion of at least 2000 lin. ft. per min. As the fumes are heavy, the exhaust vent should be at the back of the booth and on a level with the face of the operative. If a second exhaust opening is required on large work, a down pull through a bottom grating will be found more satisfactory than an overhead equipment.

A balanced supply of fresh air should be admitted to the lacquering department. This should pass over steam coils in winter, so as not to unduly lower the temperature of the room.

### WASHING, TOILET, AND DRESSING-ROOM FACILITIES

Standards have been adopted for washing, toilet, and dressing-room facilities by the New Jersey Department of Labor.

## DISCUSSION

JESSE L. JONES, \* Pittsburgh, Pa.—I would like to ask Miss Erskine if down-draft has been used in any of the foundries where heavy fumes have to be removed, instead of the overhead draft?

L. ERSKINE.—The system of overhead draft to remove heavy fumes has been very much more successful. The entire efforts of the New Jersey Department of Labor in regard to stabilizing conditions in the foundry are the result of the physical examination of the foundryman himself on first-hand information as to the prevalence of the minor ailments and the bad timekeeping that naturally accompanies them. These ailments are a factor in not only increasing the labor turnover but in increasing the percentage of bad work as well. The physical exertion of the foundryman himself is of course an important factor. In certain standard shops the amount of energy expended runs from 11 to 13 tons of foot-pounds of energy a day. In certain shops on piece work, I found a record of the energy expended per day to run as high as 22 tons. In one shop, during the war period, the management established a bonus system, and the men increased their output from 60 to 100 molds a day. The result was an increase of some 60 per cent. in output, and an increase of 400 per cent. in defective castings.

The question of the physical exertion in the unsanitary foundries, of course, is very much complicated by other factors, for which reason the department has devoted a good deal of attention to estimating what is a reasonable exertion in a sanitary, well ventilated foundry. We have met certain physical difficulties in the brass foundries that the iron foundries did not have. All that information is available through our Bureau of Industrial Information. The question of the physical energy exerted by the molder is found to have a very distinct bearing on the question of contracts, for the possibility of increasing production is balanced by the extra overhead involved.

JESSE L. JONES.—I would like to ask if any evidence has been noticed of carbon-monoxide poisoning among the employees of the foundries.

L. ERSKINE.—I have had some very interesting cases of carbon-monoxide poisoning among the employees. These, however, were due to an incidental condition, which was rapidly corrected. But we have all had the experience that during the last 3 years the procuring of material and men, and the general question of upkeep, is not on a standard basis, so that in many instances the question of establishing a natural ventilating system, by even so crude a method as knocking out a section

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\* Westinghouse Elec. & Mfg. Co.

of the wall, has been necessary. In some instances this has produced much more satisfactory results, I must admit, than some of the mechanical ventilating systems that have been devised by inexperienced engineers.

G. H. CLAMER,\* Philadelphia, Pa.—I should like to ask Miss Erskine if there have been any installations directly connected with the molds, for exhausting the atmosphere, as compared with exhausting the entire atmosphere of the foundry? In the foundry with which I am connected we pour our molds, as they do in most foundries, over the whole floor and a great many of our alloys are of very high content; therefore it would be very desirable if we could get some means of exhausting the atmosphere just at the point at which the metal is poured. We have never been able to figure out an arrangement that has been satisfactory.

L. ERSKINE.—To install a ventilating system directly over the pouring flow has proved very satisfactory. It has been done by dropping a metal curtain, which practically surrounds the pouring area; and by installing an auxiliary fan in the monitor, it has been found that the fumes are readily started in the direction desired. The trouble with most of the monitors is that they have been used for lighting purposes, and that where the width exceeds 20 per cent. of the foundry's width, they have given unsatisfactory results. Dropping the metal curtain, with a narrow enough monitor in which the windows are properly regulated, I think will give very satisfactory results.

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\* First Vice-president and Secretary, Ajax Metal Co.

### Volatility of Constituents of Brass\*

BY JOHN JOHNSTON†, ST. LOUIS, MO.

(Milwaukee Meeting, October, 1918)

THE actual amount of any metal volatilized from a mixture of metals depends on the magnitude of its partial vapor pressure under the specified conditions of temperature, and on the circumstances attending the heating—in particular, on the duration of heating, the volume of gas coming into actual contact with the surface of the metal, the extent of this surface, and the rate of diffusion of the volatile metal from the body of the melt toward the surface.<sup>1</sup> If two (or more) metals are volatilizing from the same mixture of metals, the several conditions are identical except for the difference in partial pressure and in rate of diffusion of the volatilizing metals; the second of these two factors is without doubt only of secondary importance as it is probable that the rates of diffusion of the volatilizing metals would be very similar. Hence, in order to gain an estimate of the relative amounts of two metals that may be volatilized from a given mixture, it is necessary to know the vapor pressure, or, more precisely, to ascertain for each metal its partial vapor pressure in equilibrium with the liquid (or solid) alloy under the particular circumstances. The partial pressure of each constituent depends on the effective concentration of that constituent in the alloy<sup>2</sup> as well as on the vapor pressure of the constituent when pure, and it can be calculated when both of the above factors are known. The first step, therefore, is to ascertain the vapor pressure of the several pure metals.

#### VAPOR PRESSURE OF PURE METALS

The experimental data on the vapor pressure of the several pure metals with which we are here concerned are not so accurate as could be wished, nor have the determinations been made in general at temperatures such that the results can be used directly. In a previous

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\* This paper was first printed in the *Journal* of the American Institute of Metals, March, 1918.

† Research Department, American Zinc, Lead, and Smelting Co.

<sup>1</sup> Possibly also on the latent heat of vaporization and the rate of gaseous diffusion, but these would also be subsidiary factors.

<sup>2</sup> Presumed homogeneous. If the metals are present merely as a mechanical mixture, each will show the vapor pressure corresponding to its particular degree of purity.

paper<sup>3</sup> the various data were discussed and collated, and it was found that the following formulas suffice to reproduce the experimental values and so represent the vapor pressure curve as well as it is now determined:

$$\begin{aligned}\text{For cadmium, } \log p &= -5,460 \div T + 8.04 \\ \text{For zinc, } \log p &= -6,290 \div T + 8.14 \\ \text{For lead, } \log p &= -9,900 \div T + 8.05 \\ \text{For copper, } \log p &= -16,400 \div T + 9.14 \\ \text{For iron, } \log p &= -17,000 \div T + 9.10\end{aligned}$$

in which  $p$  = vapor pressure expressed in millimeters mercury;  $T$  = absolute temperature (*i.e.*,  $t + 273$ , where  $t$  is the temperature in degrees centigrade). The accuracy of values calculated from the above formulas is of course limited by the uncertainty attaching to the original data; for cadmium and zinc the experimental data extend over a wide range so the curve is sufficiently accurate for all present purposes, but for the other metals one can be sure of little more than that the calculated values are of the right order of magnitude. On the basis of the foregoing expressions, the vapor pressure of the several pure metals at a series of temperatures is as given in Table 1.

TABLE 1.—*Vapor Pressure of Metals at Various Temperatures*

Temperature		Vapor Pressure, Expressed in mm. Mercury				
$^{\circ}\text{C}$	$T$	Cadmium	Zinc	Lead	Copper	Iron
950	1223	3800	1000	0.9	$5.4 \times 10^{-5}$	$1.6 \times 10^{-5}$
1000	1273	5600	1590	1.9	$1.8 \times 10^{-4}$	$5.6 \times 10^{-5}$
1050	1323	8100	2450	3.7	$5.6 \times 10^{-4}$	$1.8 \times 10^{-4}$
1100	1373	11500	3650	7.0	$1.6 \times 10^{-3}$	$5.3 \times 10^{-4}$
1150	1423	16000	5250	12.3	$4.2 \times 10^{-3}$	$1.4 \times 10^{-3}$

From inspection of Table 1, it is evident that whereas the individual vapor pressures increase rapidly with the temperature, the ratio of the vapor pressures of any pair does not vary much; as is indeed obvious from comparison of the formulas. For instance, the ratio of the vapor pressure of zinc to that of cadmium between  $500^{\circ}$  and  $1000^{\circ}$  ranges from about 1:7 to 1:4, and so the degree of separation achieved would be substantially independent of the temperature at which distillation of a mixture of specified composition is conducted. Accordingly, in attempting to separate two metals by fractional distillation, one would choose the temperature that is for other reasons most convenient, because relatively little is gained merely by changing the pressure, and hence the temper-

<sup>3</sup> *Journal of Industrial & Engineering Chemistry* (1917) **9**, 873; see also J. H. Hildebrand, *Journal, American Chemical Society* (1918) **40**, 45.



ature.<sup>4</sup> In this connection, it may be remarked that nothing is gained by changing the pressure of air (or other indifferent gas) upon the liquid, because the influence of pressure exerted in this way is in such cases negligible; this is stated here specifically because assertions implying the contrary are occasionally encountered.

The figures presented in Table 1, or the formulas, represent the vapor pressure of each of the metals in a pure state and, consequently, give the maximum value that can be observed at each temperature. The vapor pressure will also attain this maximum value whenever the metal is present as a pure phase in a mixture—in other words, where it is present in excess of its solubility in the mixture. This may be the case with lead even when comparatively small quantities of it are present in a brass. But when a metal is present in a homogeneous mixture, its partial vapor pressure will always be less than that of the pure metal at the same temperature, in accordance with the well known fact that the vapor pressure of a substance is lowered when a second substance is dissolved in it.

The partial vapor pressure of a metal in solution depends on its actual concentration or, better, on its effective mole fraction, in the solution. When  $1/n$  of the total moles present are moles of  $X$ , the mole fraction of  $X$  is  $1/n$ , and the partial pressure of the molecular species  $X$  is  $1/n$  of that of pure  $X$  (in which the mole fraction of  $X$  is, by definition, unity) at the same temperature. In making use of this rule it is to be borne in mind that the mole fraction of  $X$  is not necessarily given by the gross proportion of  $X$  (e.g. as determined by chemical analyses) in the mixture; for part of it may be present in other molecular species, e.g., as a compound. As an example, if the molecular proportion of zinc present in solution in a homogeneous mixture were 50 per cent. or 10 per cent., the partial vapor pressure of zinc would be one-half or one-tenth of that of pure zinc at the same temperature; but if in a homogeneous solution made by mixing 50 moles of zinc and 50 moles of a second metal, 25 of each have united to form a compound, the total number of moles is reduced to 75, the mole fraction of each molecular species is one-third, and accordingly the partial pressure of zinc is only one-third of that of pure zinc at the same temperature. The rule given has been shown to hold for binary alloys of which mercury is one constituent,<sup>5</sup>

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<sup>4</sup> If, however, the two metals form a compound that ceases to be stable above a certain temperature, one would gain by carrying out the process above that temperature; likewise if the metals are not completely miscible at all temperatures it would be advantageous to choose the temperature where the mutual solubility is least, consistent with the practical carrying out of the distillation.

<sup>5</sup> See papers by J. H. Hildebrand, *Journal*, American Chemical Society (1913), **35**, 501; *Ibid.*, (1916) **38**, 1452; J. H. Hildebrand and E. D. Eastman: *Ibid.* (1914) **36**, 2020; (1915) **37**, 2452.

it has not been demonstrated for the case under discussion, but there is every reason to believe that the cases are analogous and that its application to brasses therefore will yield results which are at least approximately true.

### PARTIAL VAPOR PRESSURE OF ZINC FROM BRASS

The experimental data bearing on this have been brought together by Gillett,<sup>6</sup> and are given in Table 2. The first column gives the gross

TABLE 2.—*Partial Vapor Pressure of Zinc from Brass*

Percentage of Zinc in Brass	Temperature Centigrade $t$	Partial Pressure of Zinc from Brass at $t$ $P$ Atm.	Vapor Pressure of Pure Zinc at $t$ $P'$ Atm.	Ratio $P/P'$ $y$	Observer
15	1365	1	32.4	0.031	Lohr
18	1300	1	22.4	0.044	Popoff
19	1300	0.95	22.4	0.042	Hansen
24	1000	0.29	2.09	0.14	Hansen
24	1084	0.66	4.26	0.15	
24	1150	1.18	6.90	0.17	
25	1220	1	11.2	0.089	Lohr
34	1110	1	5.13	0.195	Lohr
44	1050	1	3.24	0.31	Lohr
45	900	0.24	0.79	0.30	Hansen
45	950	0.44	1.31	0.34	
45	1000	0.72	2.09	0.34	
45	1100	1.55	4.80	0.32	

percentage of zinc in the several brasses; the second and third, the corresponding temperatures and partial pressures of zinc, the uncertainty in the temperatures being about  $\pm 20^\circ$ , according to Gillett; the fourth shows the vapor pressure of pure zinc at each of the several temperatures, as calculated by means of the formula given earlier; and the fifth, the ratio  $y$  of the partial pressure of the brass to that of pure zinc. This ratio also represents the mole fraction of free zinc in solution, or the net proportion of zinc present, provided that Raoult's law holds in this case. In only two of the eight brasses studied were measurements made at more than one temperature; but these two series show that the value of the ratio  $y$ , for a given brass, is unaffected, within the error of the experiment, by change of temperature. This circumstance enables us to take mean values of  $y$  and compare them with the corresponding gross proportion ( $x$ ) of zinc present in the several brasses; as is done in Table 3.

<sup>6</sup> H. W. Gillett: Brass Furnace Practice in the United States. U. S. Bureau of Mines Bull. 73 (1914), 126-8. Hansen's data are cited by Gillett from *Transactions*, American Institute of Metals (1912); the others had not been published.

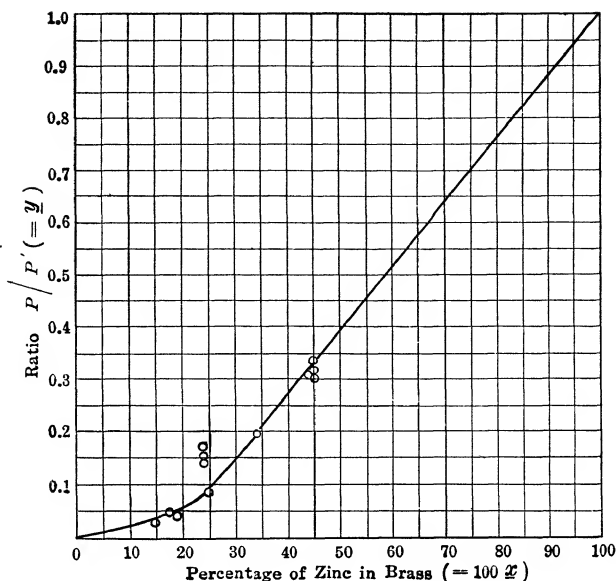


FIG. 1.—THE RATIO OF THE PARTIAL PRESSURE OF ZINC FROM BRASS TO THE VAPOR PRESSURE OF PURE ZINC AT THE SAME TEMPERATURE, FOR DIFFERENT PROPORTIONS OF ZINC IN THE BRASS.

TABLE 3.—Comparison of Gross and Net Proportions of Zinc in Brasses

Mole Fraction of Zinc *		Ratio $x/y$
Gross * $x$	Net† $y$	
0 15	0 03	5 0
0 18	0.04	4.5
0 19	0 04	4.7
0 24	0 15	
0 25	0 09	2 8
0 34	0 19	1.8
0 44	0 31	1 4
0 45	0 33	1 4

\* The atomic weights of copper and zinc are so nearly alike that the proportion by weight and mole fraction are, for present purposes, identical.

† Interpreted as such on the basis that Raoult's law holds.

In the first place, it is evident that the values of  $y$ , with one exception, increase regularly as  $x$  increases. The circumstance of this exception to the regular order points to an error in the data, presumably in the stated composition of the particular brass studied by Hansen, because the rest of his data fall in line. Moreover when the values of  $x$  are

plotted against those of  $y$ , the graph is practically linear for values of  $x$  greater than 0.2, and, when produced, passes through the point  $x = y = 1$ ; as it should do, for the gross and net proportion of zinc are then identical.

In the second place,  $y$  is smaller than  $x$ , though they approach each other and become equal at unity; in other words, of the total zinc in ordinary brasses, only a portion is, on this basis, present in solution as zinc, the remainder as a compound with copper. For example, in a

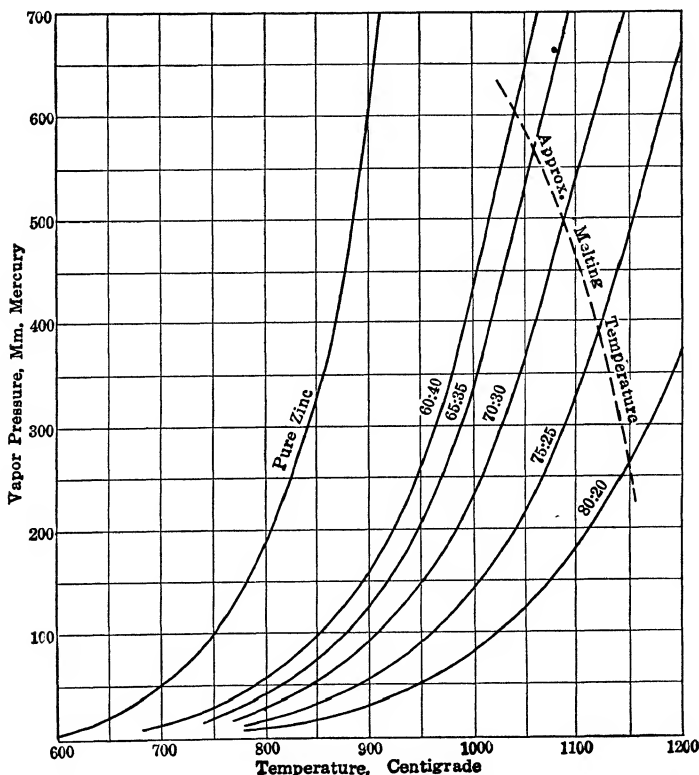


FIG. 2.—APPROXIMATE PARTIAL PRESSURE OF ZINC FROM SEVERAL BRASSES AT VARIOUS TEMPERATURES.

70:30 brass, half the zinc is "free," half is present as a compound; in a 60:40 brass, about two-thirds is "free" and one-third as a compound with copper. Moreover, a calculation from the results indicates that the compound contains not more than one mole of copper to each mole of zinc. These conclusions hold, of course, only in so far as Raoult's law is valid in this case.

In the third place, the regularity of the relation between  $x$  and  $y$  enables us to derive, by interpolation, the value of  $y$  corresponding to any given brass; and hence to calculate the partial vapor pressure

of zinc in equilibrium with that brass at any temperature. For example, for a 70:30 brass,  $y = 0.15$  and hence the partial vapor pressure is 15 per cent. of that of pure zinc at the same temperature. The results of such calculations for four typical brasses are brought together in Table 4; in connection with which it may be remarked that these results are no more uncertain than are the experimental data on which they are based, as this is merely a method of interpolation and is not dependent upon the validity of Raoult's law. It will be noticed that values are given for temperatures at which the brass is solid; in doing this we neglect the slight<sup>7</sup> change in direction of the vapor pressure curve at the melting point, a circumstance which need not be taken into account until more exact experimental observations are available.

TABLE 4.—*Approximate Partial Pressure of Zinc from Several Brasses at Various Temperatures*

Temperature Centigrade	Pure Zinc Vapor Pressure mm.	60:40 $y = 0.27$ mm.	65:35 $y = 0.21$ mm.	70:30 $y = 0.15$ mm.	80:20 $y = 0.05$ mm.
400	0.06	0.016	0.012	0.009	0.003
600	8.5	2.3	1.8	1.3	0.4
800	190	51	40	29	9.5
900	600	160	125	90	30
1000	1590	430	330	230	80
1100	3630	980	760	540	180
1200	7400	2000	1550	1100	370

The figures in Table 4 are not to be accepted as final, but at least they are of the right order of magnitude. This is corroborated by the fact that they are in harmony with the data on the volatilization of zinc from brass.<sup>8</sup> For example, we conclude that a 70:30 brass exhibits a partial pressure of about 90 mm. at 900°, which would correspond to a fairly rapid loss of zinc at that temperature, as Bengough and Hudson<sup>9</sup> found; even at 550° the partial pressure is about 0.5 mm., and so corresponds to the appreciable rate of volatilization recorded by Bassett.<sup>10</sup> Table 5 gives the numerical data on which the curves shown in Fig. 2 are based. The curve shown in Fig. 1 is based on the numerical data given in Table 2.

It is of interest to compare the partial pressure in equilibrium with the various brasses at their pouring temperatures; if we assume, with

<sup>7</sup> Slight, because the heat of melting is small in comparison with the heat of vaporization.

<sup>8</sup> Brought together by Gillett, *loc. cit.*, 129-32.

<sup>9</sup> *Journal*, Institute of Metals (1910) 4, 101.

<sup>10</sup> *Journal of Industrial & Engineering Chemistry* (1912) 4, 164.

TABLE 5.—Numerical Data on Which Curves in Fig. 2 are Based

$p$ , in Mm., for.	Temperature, Degrees Centigrade							
	600	700	750	800	900	1000	1100	1200
Zinc.....	8.5	50	100	190	600	(1590)	(3630)	(7400)
60 : 40 ( $y = 0.27$ ).....		13	27	51	160	430		
65 : 35 ( $y = 0.21$ ).....		10	21	40	125	330	760	
70 : 30 ( $y = 0.15$ ).....			15	29	90	230	540	
75 : 25 ( $y = 0.09$ ).....				17	54	140	330	670
80 : 20 ( $y = 0.05$ ).....				10	30	80	180	370

Gillett, that the latter is  $150^{\circ}$  above the melting point of the alloy, and calculate the pressures as above, we obtain the following figures:

Composition of brass.....	60:40	65:35	70:30	80:20
Pouring temperature, degrees .....	1040	1060	1090	1150
Partial pressure, mm.....	590	540	500	250

On this basis there is more danger of loss of zinc in pouring the higher zinc alloys, provided that other conditions are the same. Similar calculations can readily be made for other conditions; the above suffice to show the general relations.

In all the preceding calculations of the partial pressure of zinc from brass, we have considered, on the basis of the somewhat uncertain experimental data, that for a given brass the ratio  $y$  is independent of the temperature. The fact of such constancy, however, implies that in a given brass the proportion of zinc combined is constant, which is unlikely to be true generally; so that it is probable that  $y$  does in fact change somewhat with the temperature. The variation of  $y$  could only be established by a more careful experimental investigation of this question; such a study would constitute a new mode of attack on the question of the constitution of brass and, if properly carried through, might well yield results of importance to the whole brass industry. The necessary experimental work would not be difficult to carry out with the needful accuracy. Perhaps the most essential condition to be observed, apart from accurate determination of the actual temperature and vapor pressure,<sup>11</sup> is that the vapor space be kept small; for by this means one ensures that the actual amount of zinc volatilized is very small, hence that the composition of the brass at the vaporizing surface remains what it is presumed to be.

If such an investigation were undertaken, it would be of interest to ascertain if the ratio  $y$  for a given brass is dependent on the previous

<sup>11</sup> It would be advisable to measure the pressure directly; this can be achieved by one of the forms of glass spiral manometer (see, for instance, Preuner and Brockmüller, *Zeitschrift für Physicalische Chemie* (1913) **81**, 167.

history of the sample—for instance, whether it has been cooled very rapidly or comparatively slowly. For if, for a given brass,  $y$  changes, there must also be a change in the dissociation equilibrium between compound (or compounds) and components, with change of temperature. Now the adjustment of equilibrium to changing conditions usually requires some time, and so the actual constitution of a metal in general depends on: (a) the temperature at which it has been held, (b) whether the adjustment of the equilibrium has been complete or only partial during the cooling. Consequently, if the rate of adjustment is small, the properties of the metal will vary with the rate at which cooling takes place, by reason of differences in the proportion of free and combined zinc present in it; and these differences will be evident as differences in partial pressure of zinc depending on the previous treatment to which the brass has been subjected. It would lead too far to pursue this matter here; suffice it to state that analogous cases in organic chemistry are well known, and that the specific heat of cast metallic sodium may differ by as much as 2 per cent. according as the metal has been slowly cooled (and therefore, annealed, and in equilibrium) or rapidly chilled.<sup>12</sup>

Before leaving this aspect of the subject, I would like to express the opinion that a careful investigation of the partial pressure of zinc in equilibrium with brass, over a range of compositions and temperatures, would aid in elucidating the constitution of brass, and so be of practical value to the whole brass industry. Such a study could be undertaken and supported most suitably by some form of coöperative organization within the industry; and this is only one out of a large number of problems, the solution of which would benefit the whole industry. Indeed a coöperative organization for research is almost a necessity to the progress and vitality of an industry, particularly in view of impending conditions; and there can be little doubt that such an organization, conceived and established on broad lines, would directly repay the brass industry; for the magnitude of its operations is so large that a very slight gain in efficiency of control and in reduction of waste would soon more than offset all the expense involved. Plans of this kind are under way in Great Britain, where they are being encouraged and aided financially by the government, in Canada, Australia, New Zealand, and South Africa; and there is little, if any, less need of similar organization in this country if its industry is to compete successfully in the future.

#### VOLATILIZATION OF OTHER CONSTITUENTS

With respect to the volatilization of the other constituents of brass one cannot make very definite statements because the available data are somewhat uncertain and incomplete; but it has been thought desirable

<sup>12</sup> E. Griffiths, *Proceedings*, Royal Society of London (1914) A, **89**, 561.

to include here a brief statement about each of the several metals. The conclusions given are to be regarded as provisional; further work will be necessary to establish them.

*Copper.*—The vapor pressure of pure copper at  $1200^{\circ}$  is about 0.01 mm., and its partial pressure in equilibrium with a brass at  $1200^{\circ}$  would be less than this. Accordingly the loss of copper by volatilization would be very slight, indeed negligible for all practical purposes.<sup>13</sup>

*Iron.*—The vapor pressure of iron is so low, and the proportion of it likely to be present in a brass is so small, that no appreciable quantity of iron would volatilize from a brass under any conditions likely to be encountered in practice.

*Lead.*—In so far as the proportion of lead present in a brass is in excess of its solubility, lead will volatilize fairly rapidly,<sup>14</sup> but when the solution is homogeneous, the partial pressure of the lead should be small. For example, the partial pressure of lead in equilibrium with a brass at  $1100^{\circ}$  carrying 0.3 per cent. lead would be less than 0.1 mm., provided that the system is then homogeneous; on this basis therefore it would appear that the volatilization of lead from brasses containing only small quantities should not be rapid.

*Cadmium.*—The vapor pressure of pure cadmium is about 3.3 times that of pure zinc at temperatures about  $1000^{\circ}$ ; therefore the partial pressure of cadmium in zinc containing 0.3 per cent. cadmium (equivalent to a mole fraction of 0.0016) is  $0.0016 \times 3.3$ , or  $\frac{1}{200}$  of that of the zinc. Accordingly, since for present purposes we may assume that the number of moles of each metal lost by volatilization is proportional to its partial pressure, the ratio of cadmium to zinc lost initially would be 1 mole to 200, or about 1 to 100 by weight; and this ratio would diminish progressively owing to the relatively more rapid impoverishment of the mixture in cadmium than in zinc. Let us, however, assume that the ratio remains constant and that 100 gm. of zinc lose 5 gm. in a certain time at  $1000^{\circ}$ ; in the same period the loss of cadmium would be  $\frac{5}{100}$  or 0.05 gm. The total quantity originally present was 0.3 per cent. or 0.3 gm., so that there would remain 0.25 gm. or about 0.25 per cent. cadmium. Cadmium could therefore not be eliminated from zinc by distillation without very large losses of zinc, except by the use of a fractionating column which would be difficult to construct and operate.

When spelter carrying 0.3 per cent. of cadmium is mixed with copper to form molten brass, the relative partial pressures are still the same provided: (a) that the resulting solution is homogeneous, as we have every reason to believe is the case, (b) that each metal combines with

<sup>13</sup> Turner (*Journal, Institute of Metals* (1912), **7**, 105) observed a slight loss of copper by volatilization when a brass was heated at  $1200^{\circ}$  in a vacuum.

<sup>14</sup> Turner (*loc. cit.*) found that the lead in an alloy containing 7 per cent. of lead was distilled off at  $1200^{\circ}$  in a vacuum.



copper to about the same extent. On this latter point no information is available, but it is improbable that any difference in this respect would be great enough to change the order of magnitude of the relative partial pressures. Consequently if we assume that 5 per cent. of the zinc is lost by volatilization during the process of brass making, the amount of cadmium would be about 0.26 per cent. relative to the zinc or about 0.08 per cent. relative to the brass. On the other hand, it is established that the cadmium content of brass made from spelter carrying 0.3 per cent. cadmium is certainly less than 0.02 per cent.; a fact which shows that the actual loss of cadmium in the process of brass making is about four times as great as that which, according to the above calculations, could be ascribed to direct volatilization. This, coupled with the fact that, when a brass is heated, the ratio of cadmium to zinc diminishes very slowly, indicates that the main loss of cadmium occurs during the melting together of the constituents of the brass; and suggests that the cadmium acts as a deoxidizing agent<sup>15</sup> and is removed from the brass as oxide. This of course is little more than a surmise; but the matter could readily be elucidated by analyses to determine whether the cadmium which disappears from the brass reappears in the flue dust,<sup>16</sup> in the dross or elsewhere.

#### SUMMARY

Comparison of the measurements of the partial pressure of zinc in equilibrium with brass, with the vapor pressure of pure zinc at the same temperature, enables one to calculate preliminary values of the partial pressure of zinc in equilibrium with any brass at any temperature; these values are in harmony with the data on the volatilization of zinc from brass. A discussion of the results points to the conclusion that only a portion of the zinc exists in brass as free zinc in solution, the remainder being present as a compound with copper. A careful and systematic experimental investigation of this question would be highly desirable as it would throw new light on the constitution of brass, and so be of benefit to the whole industry; such an investigation, in the writer's opinion, could best be undertaken by coöperation between those primarily interested.

Consideration of the partial pressure of the other usual constituents

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<sup>15</sup> Presumably cadmium would tend to deoxidize copper at 1000–1100° more readily than would zinc, because cadmium oxide is more volatile than zinc oxide. It is rumored that one can materially increase the tensile strength of copper wire without serious prejudice to its conductivity by the addition of a small proportion (1 per cent. or less) of cadmium.

<sup>16</sup> Bassett, working with a spelter carrying about 0.5 per cent. cadmium found the ratio of cadmium to zinc in the flue dust to be about 4 per cent., whereas it would have been nearer 10 per cent. if all the cadmium eliminated had condensed in the flue. *Journal of Industrial & Engineering Chemistry* (1912) 4, 164.

of brass indicates that under all ordinary conditions the loss by volatilization of copper or iron is negligible, that of lead or cadmium would not be rapid. The fact that cadmium is, for practical purposes, completely eliminated in the process of brass making indicates that some factor other than volatilization is also acting; but this is a matter which will require further work before definite statements as to the mode of elimination of the cadmium can be made.

## DISCUSSION

J. W. RICHARDS\* (South Bethlehem, Pa.).—The brass industry would find it very profitable to subsidize researches on the purely scientific determination of the thermophysical properties of the brasses. The data we need most in connection with the brasses are the vapor tension of the zinc from the brasses at different temperatures, the heat of combination of zinc with copper to form different kinds of brasses, and the latent heat of fusion of these brasses at the melting temperature.

It is thermodynamically possible to take the formula for the vapor tension of zinc and calculate from it the vapor tension of the zinc from the brasses, if we know the heat of combination. The work done in volatilizing zinc alone is well known, the thermodynamic work of separating it from the copper is known for only one alloy, the 52 per cent. zinc alloy. It should be known for all the brasses. Then we could calculate the vapor tension of zinc from all the brasses in the liquid state as exactly as we know the vapor tension of zinc. If we know the latent heat of fusion of the brasses at the melting point, since the vapor tension of zinc from the solid brass at the melting point is the same as that from liquid brass, we can use that data for transforming the equation so as to give us the equation for the vapor tension of zinc from the solid brass, with mathematical certainty; also, we could in this way get the vapor tension of the zinc from the solid brasses down as low as we want to evaluate the formula—even to the ordinary temperature, as a matter of scientific curiosity.

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## A Comparison of Grain-size Measurements and Brinell Hardness of Cartridge Brass

BY W. H. BASSETT\* AND C. H. DAVIS,† WATERBURY, CONN.

(New York Meeting, February, 1919)

IN the commercial annealing of cartridge brass there are four points regarding which definite data are essential. They have to do with the correct interpretation of grain count in its relation to annealing temperature and, incidentally, to Brinell hardness. These points are:

1. The comparison of the grain sizes of two cartridge-brass mixtures: 69 copper, 31 zinc, 0.376-in. (9.5-mm.) gage; and 68 copper, 32 zinc, 0.130-in. (3.3-mm.) gage.

2. The comparison of the grain sizes of annealed metal that had previously been reduced by rolling varying amounts; for instance, 20.0, 36.6, 50.9, and 59.1 per cent.

3. The determination of standards for grain sizes on annealed brass of the composition 68 per cent. copper, 32 per cent. zinc and 69 copper, 31 zinc.

4. The comparison of grain size with Brinell hardness on identical samples of annealed metal.

In their comprehensive and thorough investigation of the recrystallization of cold-worked alpha brass on annealing,<sup>1</sup> Mathewson and Phillips have discussed the relations between temperature of anneal, degree of deformation, and structural alteration in alpha brass. They have also shown certain comparisons between the ordinary physical properties and the grain size of annealed brass. The purpose of the present investigation is mainly concerned with the grain size of cartridge brass, its relation to Brinell hardness, and the publication of sufficient data to enable those engaged in the inspection of such material to have a correct foundation upon which to work.

The first alloy was taken from regular mill stock that had been rolled from 0.580 in. to 0.376 in. (14.7 mm. to 9.5 mm.) gage, a reduction of 35.1 per cent. This bar (No. 1) had the following composition: Copper, 69.20 per cent.; zinc, 30.76 per cent.; lead, 0.02 per cent.; iron, 0.02 per cent.

The second alloy was also taken from mill stock of 0.325 in. (8.25 mm.) gage and was rolled as shown in Table 1, in order to get four bars reduced two, four, six, and eight B. & S. numbers respectively. This second alloy

\* American Brass Co.

† American Brass Co.

<sup>1</sup> *Trans.* (1916) 54, 608-657.

had the composition: Copper, 68.48 per cent.; zinc, 31.47 per cent.; lead, 0.02 per cent.; iron, 0.03 per cent.

TABLE 1.—*Results Obtained by Rolling Bars*

Bar No.	Annealed Bar Selected from Stock, Inch	Rolled to, Inch	Annealed on, Inch	Rolled to, Inch	Reduction by Rolling, Per Cent.
2	0 325	.....	0 325	0.133	59.1
3	0.325	0.273	0.273	0.134	50.9
4	0.325	0.213	0.213	0.135	36.6
5	0.325	0 168	0.168	0.134	20.2

Specimens, 1 by 3 in. (25.4 by 76.2 mm.), from each of these five bars were annealed for  $\frac{1}{2}$  hr. at 50° C. intervals, from 200° C. to 850° C. From 275° C. to 425° C., additional samples were annealed at 25° C. intervals. The specimens were tightly wrapped together in sheet copper and were quenched with their covering as quickly as possible at the end of  $\frac{1}{2}$  hr. at the required temperature. A new Bristol indicator and recorder with base-metal couple was used, the latter being wrapped with the specimens. The couple was checked and calibrated before and after annealing by the boiling point of sulfur and the melting point of sodium chloride.

The Brinell hardness tests were made with an Aktiebolaget alpha machine. A load of 500 kg. and the standard 10-mm. diameter ball were used and the pressure maintained for 30 sec. The pressure exerted by this machine was checked by weighing on a standard scale. The surfaces of both hard and annealed specimens were scoured with emery cloth and polished with fine emery before testing. Two impressions were taken, one in the center of the specimen and the other halfway toward the end. Readings of these were made upon an 80 mm. Gaertner comparator, accurate to 0.001 mm. One reading was taken in the direction of the original rolling, the other at right angles to that direction for each impression, and the four results averaged. Little or no discrepancy was found in these results, except in the case of the hard-rolled specimens where the impression was oblong, the longer diameter coinciding in direction with the direction of rolling. In the harder samples this difference in diameter was equivalent to as much as 10 Brinell points.

The grain size was counted on a section taken parallel to the surface between the two Brinell impressions. On the specimens annealed below 700° C. the magnification used was 150 diameters; at 700° C., 75 diameters; and from 750° C. to 850° C., 50 diameters. The method of counting used is recommended by the American Society for Testing Materials.<sup>2</sup>

<sup>2</sup> Tentative Definitions and Rules Governing the Preparation of Micrographs of Metals and Alloys. *Proceedings*, American Society for Testing Materials (1917) 17, Pt. 1, 838.

It is also described by Zay Jeffries and others.<sup>3</sup> A circle 79.8 mm. in diameter was used in counting and the diameter of the average grain in millimeters was determined. The following formulas, proposed by Prof. Jeffries, were used:

- $w$  = boundary grains;
- $z$  = completely included grains;
- $x$  = equivalent number of whole grains in 5000 sq. mm. (circle 79.8 mm. diameter or rectangle having area of 5000 sq. mm.);
- $m$  = magnification used;
- $f$  = multiplier to obtain grains per square millimeter;
- $n$  = number of grains per square millimeter;
- $d$  = diameter of average grain in millimeters;
- $a$  = area of average grain in  $U^2$ .

$$x = \frac{1}{2} w + z \qquad f = \frac{m^2}{5000} \qquad n = fx$$

$$d = \frac{1}{\sqrt{n}} \qquad a = \frac{1,000,000}{n}$$

Tables 2, 3, and 4 give a résumé of the results obtained.

TABLE 2.—*Brinell Hardness and Grain Size on 69-31 Brass*  
(See Fig. 2)

Bar No. 1. Rolled from 0.580 in. to 0.376 in. (14.7 mm. to 9.5 mm.).  
Reduction by rolling 35.1 per cent. (4—B. & S. numbers. Hard)

Anneal. Temp., Degrees C.	$x$	Mag. Dia.	$f$	$n$	$\sqrt{n}$	$d$	Av. $d$	Av. Brinell Number from Two Imp. and Four Readings
850	10.0	50	0.5	5.0	2.23	0.448	.....	41.3
800	24.0	50	0.5	12.0	3.4	0.3	.....	43.9
750	52.5	{ 50 75 }	{ 0.5 1.125 }	26.2	5.1	{ 0.198 0.196 }	0.197	46.0
700	73.0	{ 75 150 }	{ 1.125 4.5 }	82.12	9.06	{ 0.110 0.0587 }	0.110	50.6
650	64.5	{ 75 150 }	{ 1.125 4.5 }	290.25	17.03	{ 0.0667 0.0464 }	0.062	55.8
600	127.0	{ 75 150 }	{ 1.125 4.5 }	571.5	23.90	{ 0.0418 0.0381 }	0.044	61.7
550	226.0	{ 75 150 }	{ 1.125 4.5 }	1017.0	31.89	{ 0.0313 0.0222 }	0.033	65.9
500	{ 449.0 368.0 }	150	4.5	{ 2020.5 1656.0 }	{ 44.95 40.6 }	{ 0.024 0.0190 }	0.023	70.4
450	{ 617.0 531.0 }	150	4.5	{ 2776.5 2389.5 }	{ 52.69 48.8 }	{ 0.020 0.020 }	0.020	75.0
425	.....	...	....	.....	.....	.....	.....	75.2
400	.....	...	....	.....	.....	.....	.....	82.6
375	.....	...	....	.....	.....	.....	.....	88.3
350	.....	...	....	.....	.....	.....	.....	102.0
325	.....	...	....	.....	.....	.....	.....	102.0
300	.....	...	....	.....	.....	.....	.....	143.0
275	.....	...	....	.....	.....	.....	.....	153.0
250	.....	...	....	.....	.....	.....	.....	153.0
200	.....	...	....	.....	.....	.....	.....	153.0
Hard....	.....	...	....	.....	.....	.....	.....	146.0

<sup>3</sup> *Trans.* (1916) **54**, 594-607. Elaborated in *Metallurgical and Chemical Engineering* (Feb. 15, 1918) **8**, 185.

TABLE 3.—*Brinell Hardness and Grain Size on 68-32 Brass*

(See Figs. 1 and 3)

Bar No. 2 (Mark ●). Reduction by rolling 20.2 per cent.

(2—B. &amp; S. numbers)

Anneal. Temp., Degrees C.	$x$	Mag. Dia.	$f$	$n$	$\sqrt{n}$	$d$	Av. Brinell Number from Two Imp. and Four Readings
850	15 0	50	0.5	7.5	2.74	0.365	41.7
800	32.5	50	0.5	16.25	4.03	0.247	43.6
750	57.5	50	0.5	28.75	5.36	0.186	45.7
700	71.0	75	1.125	79.875	8.93	0.112	49.0
650	42 5	150	4.5	191.25	13.82	0.072	52.0
600	62.0	150	4.5	279.0	16.70	0.059 +	56.1
550	87.0	150	4.5	391.5	19.7	0.0507	58.2
500	110.0 ±	150	4.5	495.0	22.2	0.045	60.5
450	118.5	150	4.5	533.25	23.09	0.043	65.5
425	.....	...	.....	.....	.....	.....	69.1
400	One-fourth area consists of new grains.						89.7
375	Few new grains.						94.1
350	No new grains.						101.0
325	No new grains.						100.0
300	No new grains.						103.0
275	.....	...	.....	.....	.....	.....	104.0
250	.....	.....	.....	.....	.....	.....	106.0
200	.....	...	.....	.....	.....	.....	109.0
Hard....	.....	...	.....	.....	.....	.....	110.0

Bar No. 3 (Mark +). Reduction by rolling 36.6 per cent.

(4—B. &amp; S. numbers)

850	17.0	50	0.5	8.5	2.91	0.344	41.1
800	44 0	50	0.5	22.0	4.69	0.213	43.1
750	71 5	50	0.5	35.75	5.97	0.167	45.9
700	52.0	75	1.125	58.50	7.65	0.131	48.9
650	34.5	150	4.5	155.25	12.46	0.080	51.5
600	83.0	150	4.5	373.5	19.30	0.0518	55.4
550	142.5	150	4.5	641.25	25.32	0.0395	61.3
500	196.0	150	4.5	882.0	29.70	0.034	66.4
450	198.0	150	4.5	891.0	29.85	0.0336	70.5
425	.....	...	.....	.....	.....	.....	71.2
400	Completely recrystallized.						74.6
375	.....	.....	.....	.....	.....	.....	88.8
350	One-third area consists of new grains.						100.0
325	Some new grains.						106.0
300	No new grains.						137.0
275	No new grains.						140.0
250	No new grains.						143.0
200	No new grains.						148.0
Hard....	.....	...	.....	.....	.....	.....	147.0 } 142
							136.0 }

TABLE 4.—*Brinell Hardness and Grain Size on 68-32 Brass*  
(See Figs. 1 and 3)  
Bar No. 4 (Mark X). Reduction by rolling 50.9 per cent.  
(6 + B. & S. numbers)

Anneal. Temp., Degrees C	<i>x</i>	Mag. Dia.		<i>n</i>	$\sqrt{n}$	<i>d</i>	Av. Brinell Number from Two Imp and Four Readings
850	18.5	50	0.5	9.25	3.04	0.329	42.0
800	38.0	50	0.5	19.0	4.35	0.230	43.1
750	67.5	50	0.5	33.75	5.80	0.172	46.4
700	68.0	75	1.125	76.50	8.75	0.114	49.7
650	38.0	150	4.5	171.0	13.07	0.076	52.1
600	84.0	150	4.5	378.0	19.43	0.0515	56.8
550	165.5	150	4.5	744.75	27.28	0.0366	62.0
500	177.0	150	4.5	796.5	28.20	0.0355	69.1
450	446.0+	150	4.5	2007.0	44.8	0.022	74.1
425	.....	...	.....	.....	.....	.....	76.5
400	.....	.....	Completely recrystallized.				79.6
375	.....	.....	.....	.....	.....	.....	86.4
350	.....	.....	Few remnants of former crystals.				91.2
325	.....	.....	.....	.....	.....	.....	93.3
300	.....	.....	One-fourth area consists of new grains.				130.0
275	.....	.....	Few new grains.				155.0
250	.....	.....	No new grains.				166.0
200	.....	.....	No new grains.				169.0
Hard....	.....	...	.....	.....	.....	.....	163.0
							153.0 } 158

Bar No. 5 (Mark O). Reduction by rolling 59.1 per cent.  
(8—B. & S. numbers)

850	16.0	50	0.5	8.0	2.83	0.353	41.7
800	36.5	50	0.5	18.25	4.27	0.234	44.1
750	77.0	50	0.5	38.5	6.50	0.161	46.4
700	66.0	75	1.125	74.25	8.62	0.116	49.2
650	38.5	150	4.5	173.25	13.17	0.076	52.4
600	78.0	150	4.5	351.0	18.73	0.0534	57.2
550	175.5	150	4.5	789.75	28.1	0.0356	62.0
500	314.5	150	4.5	1415.25	37.5	0.0266	69.1
450	400.0	150	4.5	1800.0	42.4	0.0236	77.4
425	.....	...	.....	.....	.....	.....	79.0
400	.....	.....	Completely recrystallized				83.8
375	.....	.....	.....	.....	.....	.....	88.6
350	.....	.....	Completely recrystallized.				91.9
325	.....	.....	.....	.....	.....	.....	93.3
300	.....	.....	One-half to six-tenths new grains.				124.0
275	.....	.....	Few new grains				154.0
250	.....	.....	No new grains.				171.0
200	.....	...	.....	.....	.....	.....	172.0
Hard....	.....	...	.....	.....	.....	.....	168.0
							158.0 } 163

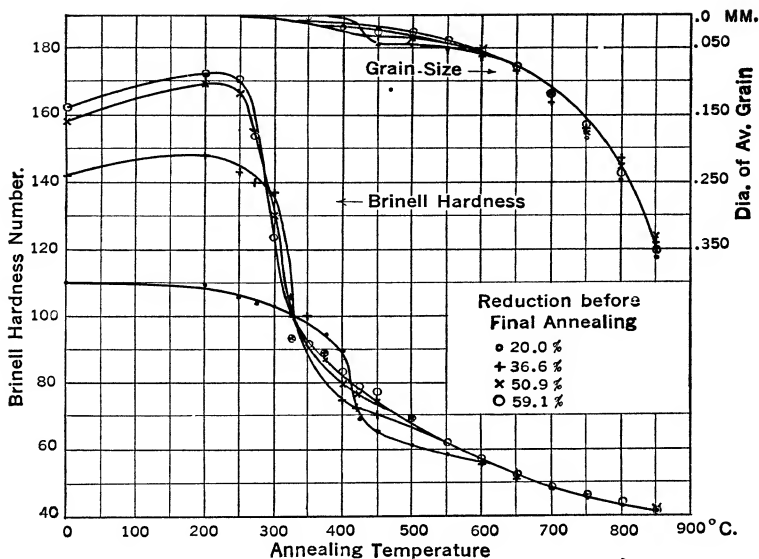


FIG. 1.—DIRECT COMPARISON OF BRINELL AND GRAIN-SIZE MEASUREMENTS ON 0.130-IN. GAGE CARTRIDGE BRASS. ANNEALED  $\frac{1}{2}$  HR. AT TEMPERATURES NOTED. SPECIMENS WERE PREVIOUSLY REDUCED BY ROLLING AS INDICATED.

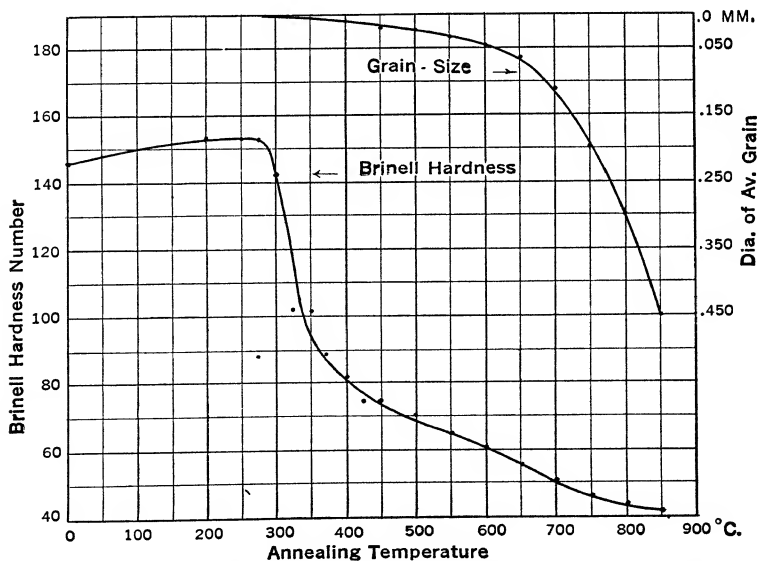


FIG. 2.—DIRECT COMPARISON OF BRINELL AND GRAIN-SIZE MEASUREMENTS ON 0.376-IN. GAGE CARTRIDGE BRASS. ANNEALED  $\frac{1}{2}$  HR. AT TEMPERATURES NOTED. SPECIMENS WERE PREVIOUSLY REDUCED 35.1 PER CENT. BY ROLLING.



The micrographs taken to illustrate this paper are magnified 75 diameters but the grain counts were made on micrographs taken in the same area but magnified 50, 75, or 150 diameters. Accompanying this report are three plots. The first, Fig. 1, shows the relation of Brinell hardness to annealing temperature and the relation of grain size to annealing temperature for the 68-32 alloy; the second, Fig. 2, shows the same relations for the 69-31 alloy, the third, Fig. 3, shows the relation of Brinell hardness to grain size for a given alloy of copper and zinc.

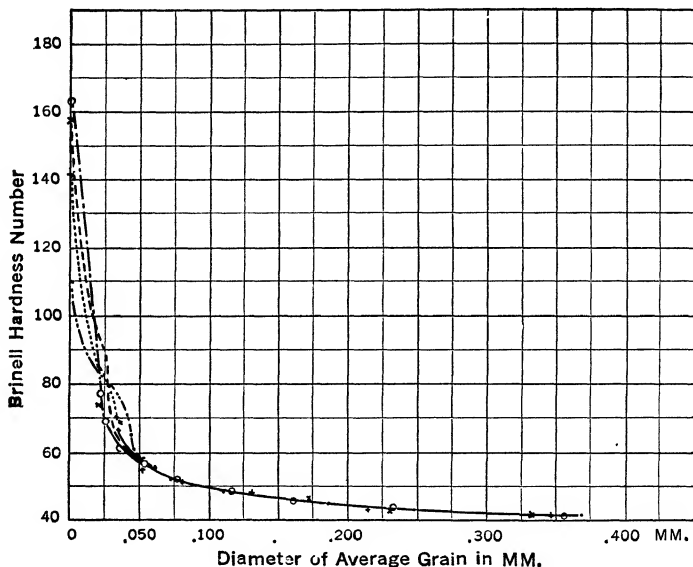


FIG. 3.—BRINELL HARDNESS INDICATES GRAIN SIZE FOR ANY GIVEN BRASS IN THE ALPHA PHASE. AT LOW TEMPERATURES THE GRAIN SIZE IS INFLUENCED BY THE GRAIN SIZE OF THE PREVIOUS ANNEAL, 650° C. IN THIS CASE. PLOTTED FROM THE DATA USED FOR FIG. 1.

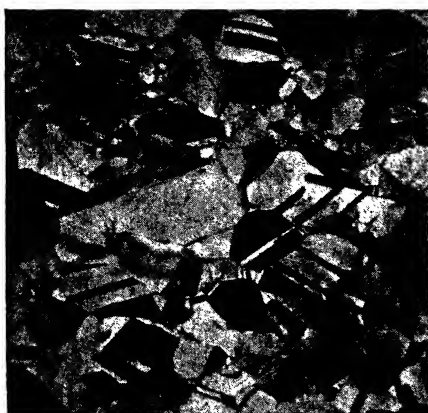
The Brinell method is a very accurate way of determining the hardness of sheet brass. The hardness of rolled metal is relatively proportionate to the percentage reduction by rolling.<sup>4</sup> The hardness of annealed metal is relatively proportionate to the temperature of annealing<sup>5</sup> (for any fixed period of time). The Brinell hardness of annealed metal is proportionate to the annealing temperature, but this proportion varies on account of two factors; namely, the amount of the last rolling the metal received, and the grain size that existed at the time of that rolling. Figs. 1 and 4 to 13 illustrate these points in the case of the 0.130-in. (3.3-mm.) gage cartridge brass and show: that in annealing hard-

<sup>4</sup> C. H. Davis: Testing of Sheet Brass. *Proceedings*, American Society for Testing Materials (1917) 17, Pt. 2, 166.

<sup>5</sup> C. H. Davis: *Op cit.*, Fig. 8 and Fig. 3.

20.2 PER CENT. REDUCTION

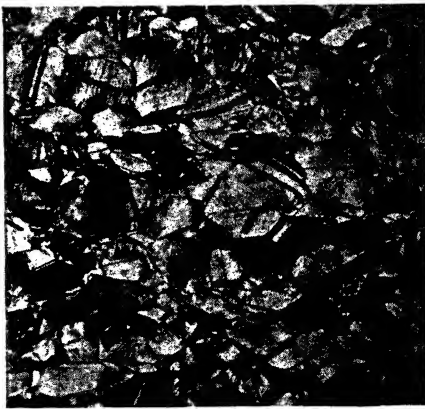
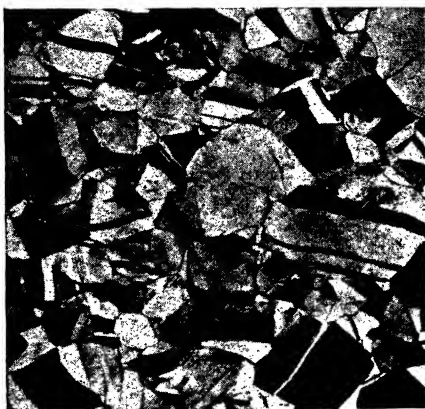
36.6 PER CENT. REDUCTION



BRIN. 110

AS ROLLED

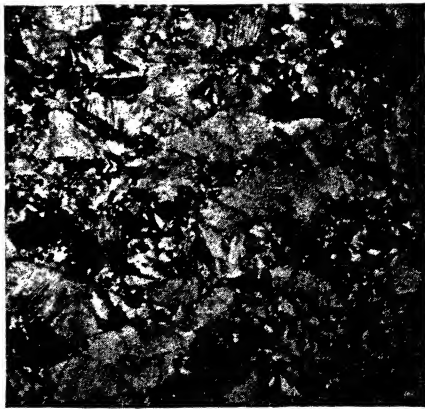
BRIN. 142



BRIN. 103

300° C. ANNEAL

BRIN. 137



BRIN. 100

325° C. ANNEAL

BRIN. 106

FIG. 4.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

50.9 PER CENT. REDUCTION



BRIN. 158

59.1 PER CENT REDUCTION



BRIN. 163

AS ROLLED

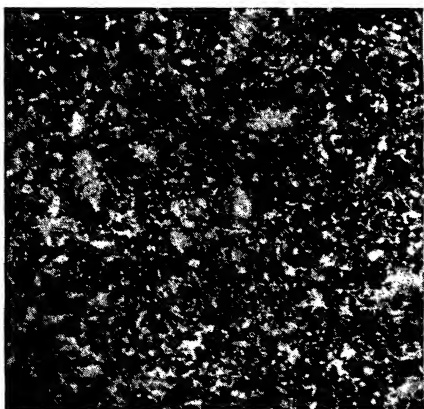


BRIN. 130

300° C. ANNEAL

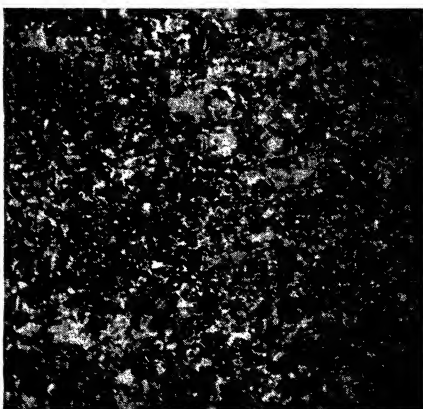


BRIN. 124



BRIN. 93.3

325° C. ANNEAL

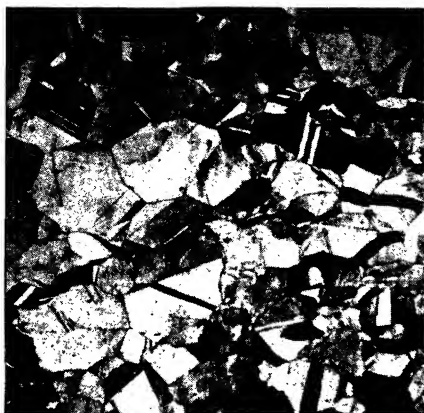


BRIN. 93.3

FIG. 5.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

20.2 PER CENT. REDUCTION

36.6 PER CENT. REDUCTION



BRIN. 101

350° C. ANNEAL

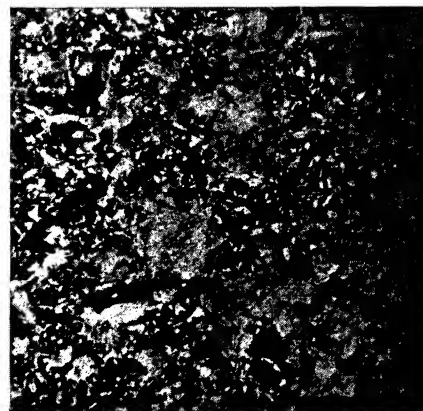


BRIN. 100

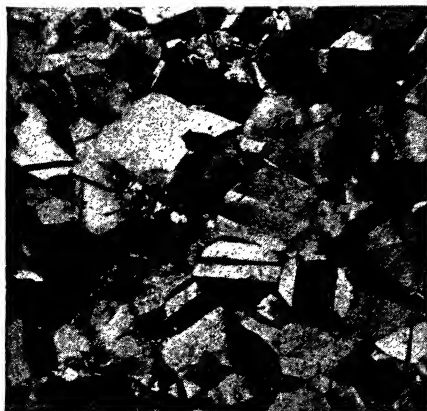


BRIN. 94.1

375° C. ANNEAL

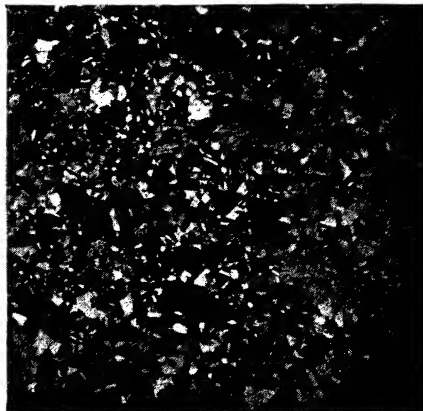


BRIN. 88.8



BRIN. 89.7

400° C. ANNEAL

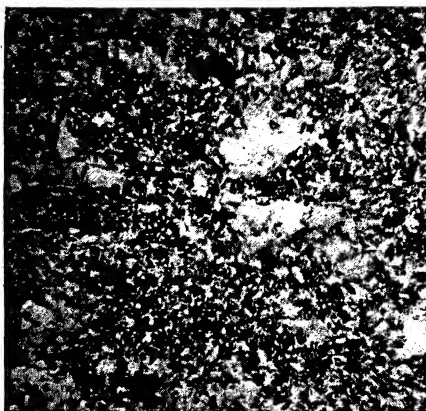


BRIN. 74.6

FIG. 6.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

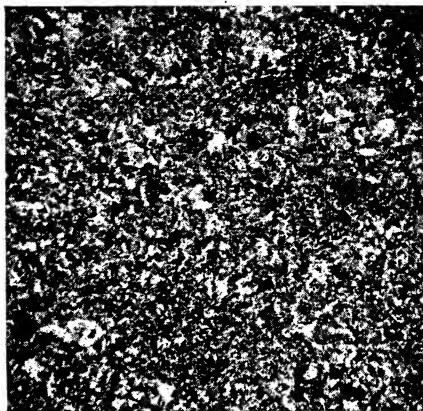
50.9 PER CENT. REDUCTION

59.1 PER CENT. REDUCTION

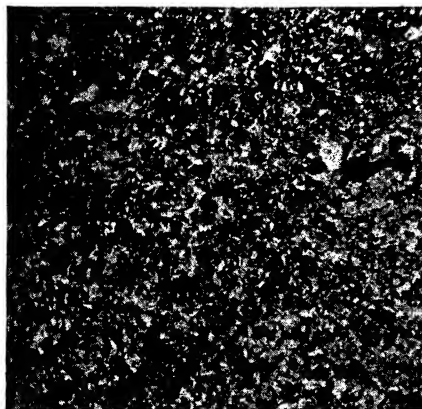


BRIN. 91.2

350° C. ANNEAL

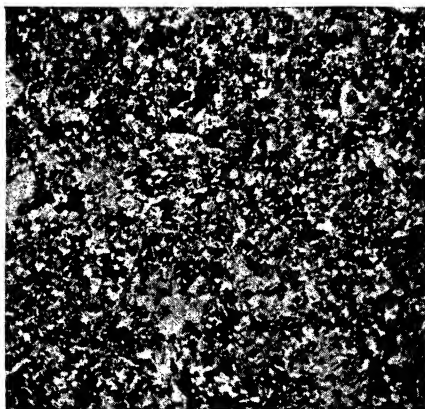


BRIN. 91.9

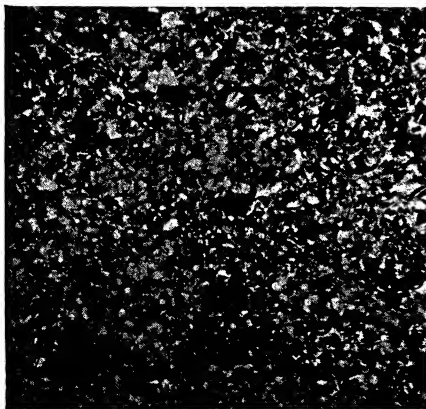


BRIN. 86.4

375° C. ANNEAL

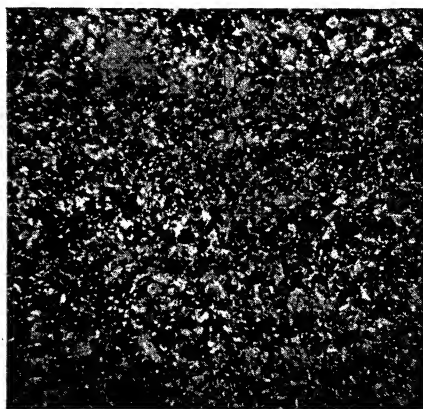


BRIN. 88.6



BRIN. 79.6

400° C. ANNEAL



BRIN. 83.8

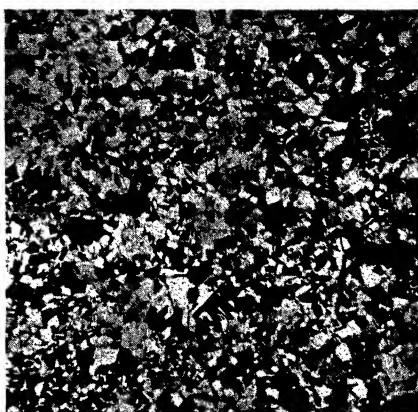
FIG. 7.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

20.2 PER CENT. REDUCTION

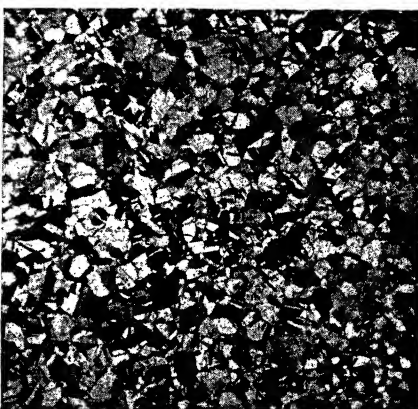
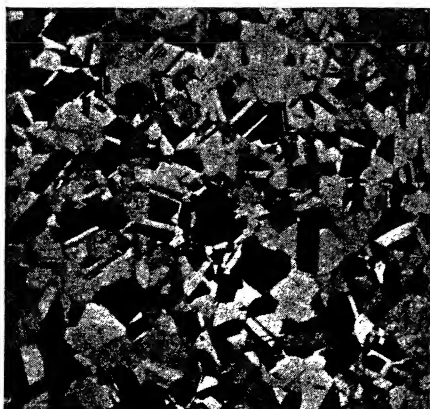
BRIN. 65.5,  $d = 0.043$  MM.

450° C. ANNEAL

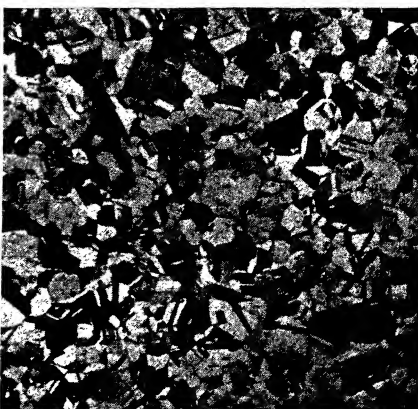
36.6 PER CENT. REDUCTION

BRIN. 70.5,  $d = 0.0336$  MM.BRIN. 60.5,  $d = 0.045$  MM.

500° C. ANNEAL

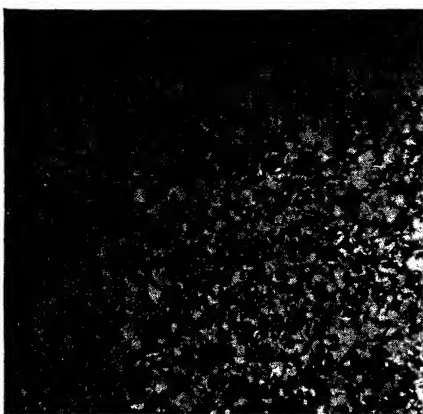
BRIN. 66.4,  $d = 0.034$  MM.BRIN. 58.2,  $d = 0.0507$  MM.

550° C. ANNEAL

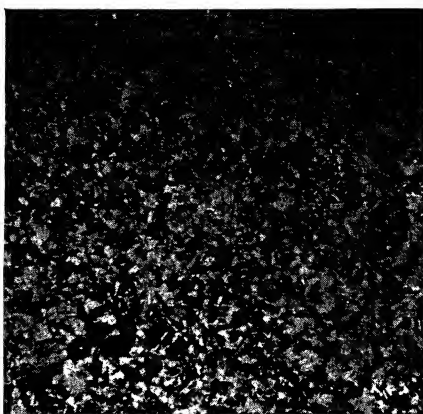
BRIN. 61.3  $d = 0.0395$  MM.FIG. 8.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .



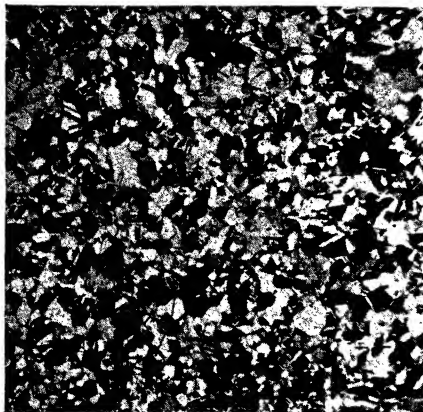
50.9 PER CENT. REDUCTION

BRIN. 74.1,  $d = 0.022$  MM.

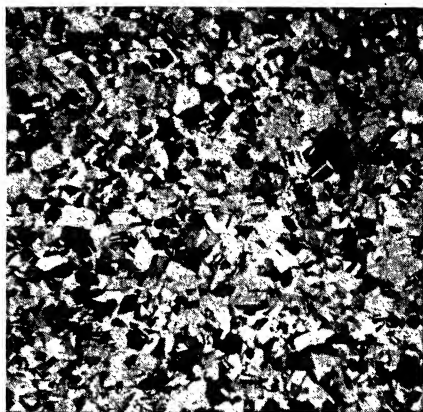
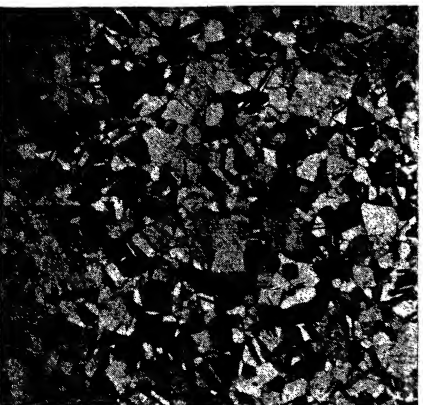
59.1 PER CENT. REDUCTION

BRIN. 77.4,  $d = 0.0236$  MM.

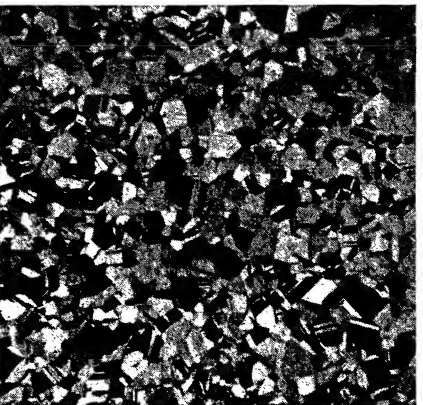
450° C. ANNEAL

BRIN. 69.1,  $d = 0.0355$  MM.

500° C. ANNEAL

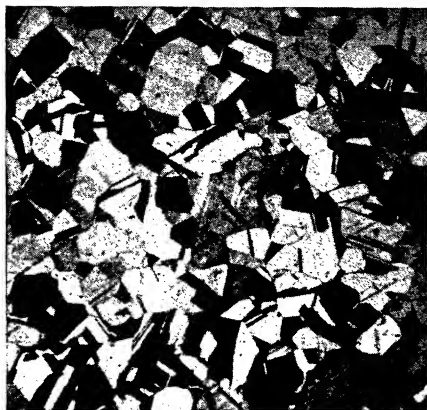
BRIN. 69.1,  $d = 0.0266$  MM.BRIN. 62.0,  $d = 0.0366$  MM.

550° C. ANNEAL

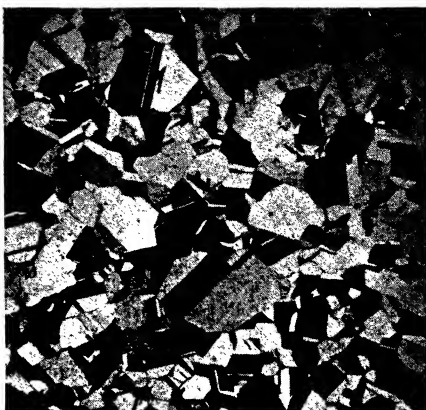
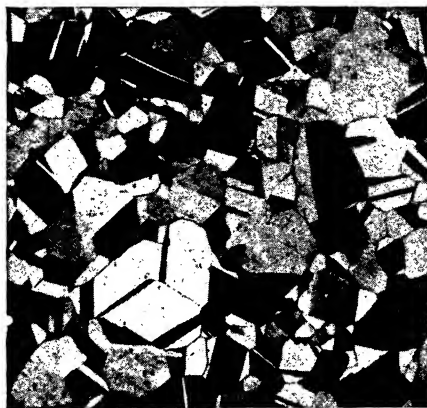
BRIN. 62.0,  $d = 0.0356$  MM.FIG. 9.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

20.2 PER CENT. REDUCTION

36.6 PER CENT. REDUCTION

BRIN. 56.1,  $d = 0.059$  MM.

600° C. ANNEAL

BRIN. 55.4  $d = 0.0518$  MM.BRIN. 52.0,  $d = 0.072$  MM.

650° C. ANNEAL

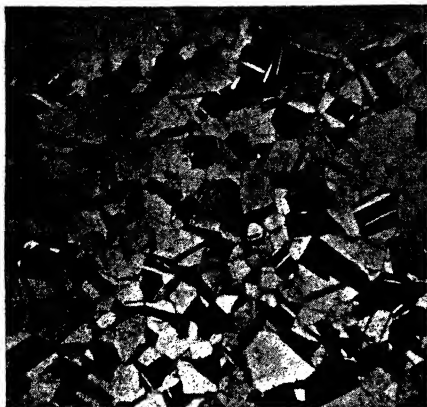
BRIN. 51.5,  $d = 0.080$  MM.BRIN. 49.0,  $d = 0.112$  MM.

700° C. ANNEAL

BRIN. 48.9,  $d = 0.131$  MM.FIG. 10.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

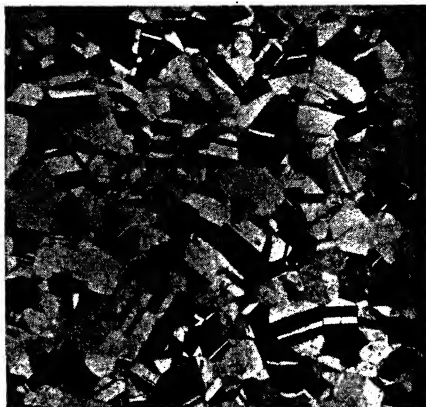
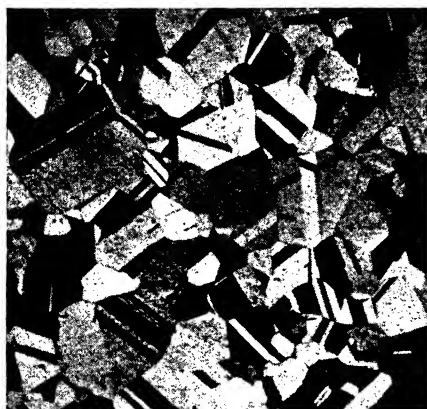


50.9 PER CENT. REDUCTION

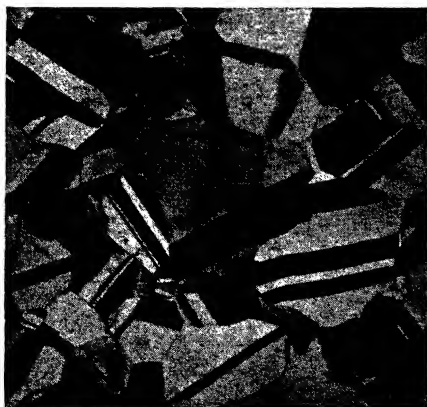
BRIN. 56.8,  $d = 0.0515$  MM.

600° C. ANNEAL

59.1 PER CENT. REDUCTION

BRIN. 57.2,  $d = 0.0534$  MM.BRIN. 52.1,  $d = 0.076$  MM.

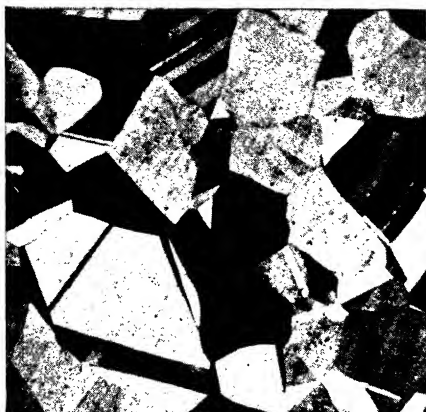
650° C. ANNEAL

BRIN. 52.4,  $d = 0.076$  MM.BRIN. 49.7,  $d = 0.114$  MM.

700° C. ANNEAL

BRIN. 49.2,  $d = 0.110$  MM.FIG. 11.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

20.2 PER CENT. REDUCTION

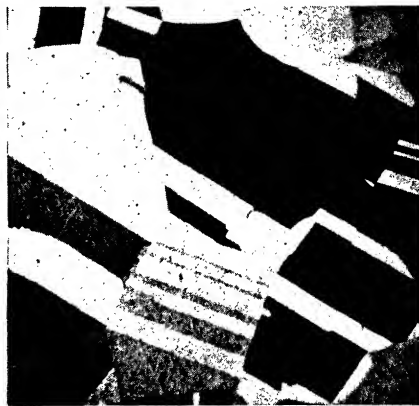
BRIN. 45.7,  $d = 0.186$  MM.

750° C. ANNEAL

36.6 PER CENT. REDUCTION

BRIN. 45.9,  $d = 0.167$  MM.BRIN. 43.6,  $d = 0.247$  MM.

800° C. ANNEAL

BRIN. 43.1,  $d = 0.213$  MM.BRIN. 41.7,  $d = 0.365$  MM.

850° C. ANNEAL

BRIN. 41.1,  $d = 0.344$  MM.FIG. 12.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .

50.9 PER CENT. REDUCTION

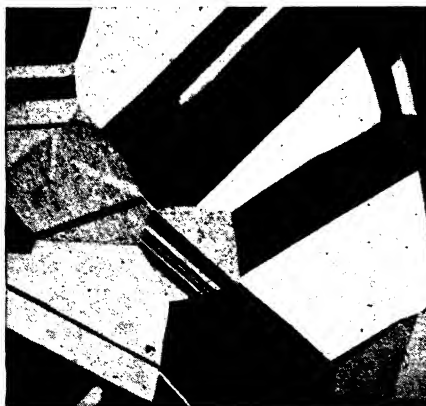
BRIN. 46.4,  $d = 0.172$  MM.

750° C. ANNEAL

59.1 PER CENT. REDUCTION

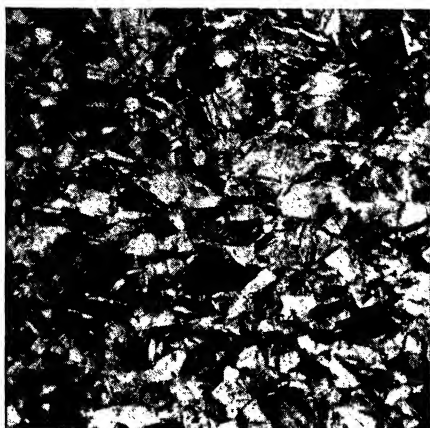
BRIN. 46.4,  $d = 0.161$  MM.BRIN. 43.1,  $d = 0.230$  MM.

800° C. ANNEAL

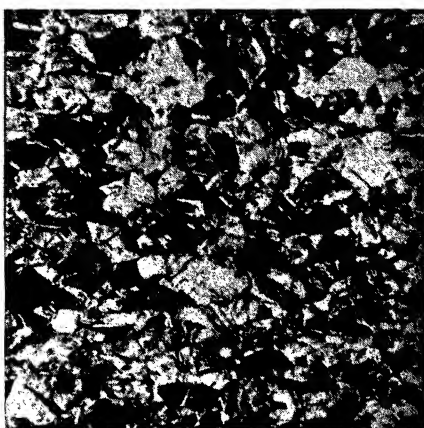
BRIN. 44.1,  $d = 0.234$  MM.BRIN. 42.0,  $d = 0.329$  MM.

850° C. ANNEAL

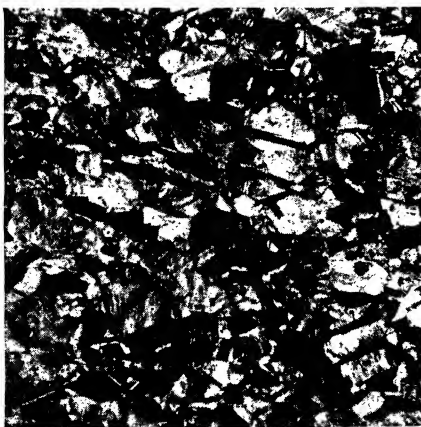
BRIN. 41.7,  $d = 0.353$  MM.FIG. 13.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .



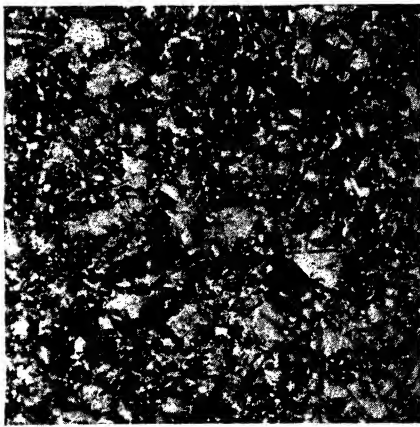
BRIN. 146—As rolled.



BRIN. 153—250° C.

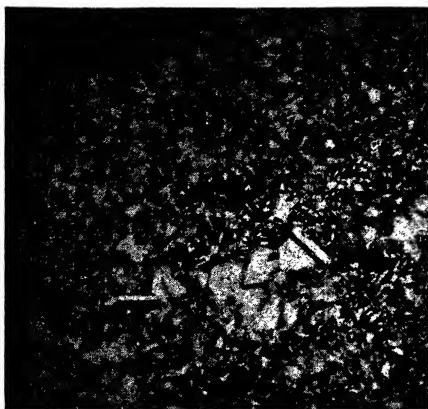


BRIN. 143—300° C.

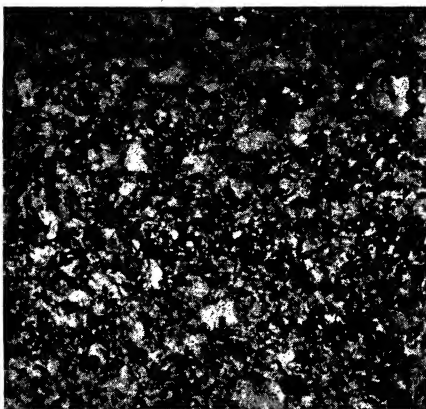


BRIN. 102—325° C.

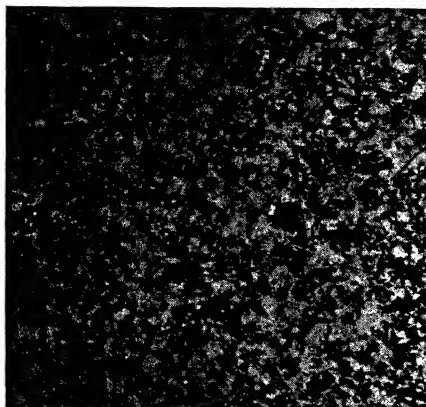
FIG. 14.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .



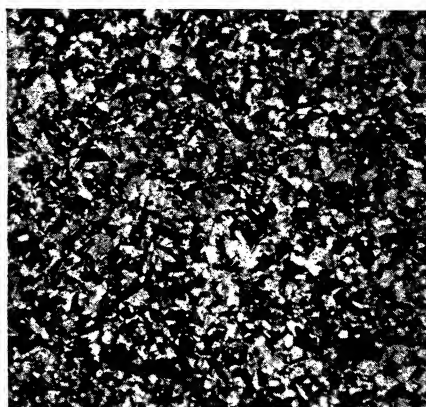
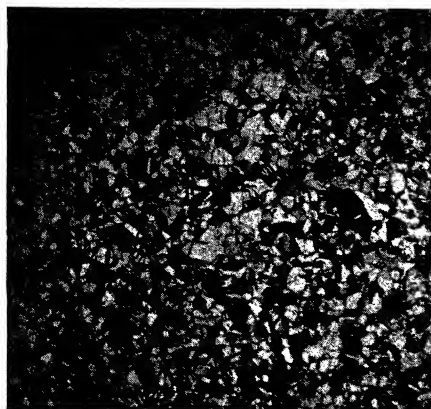
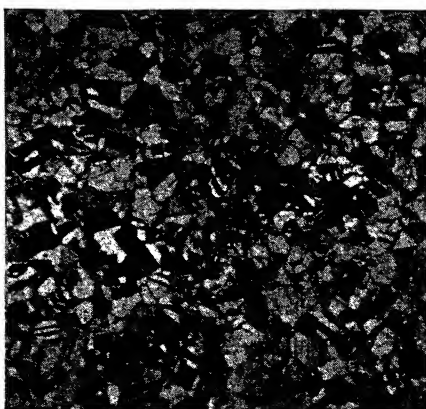
BRIN. 102—350° C.

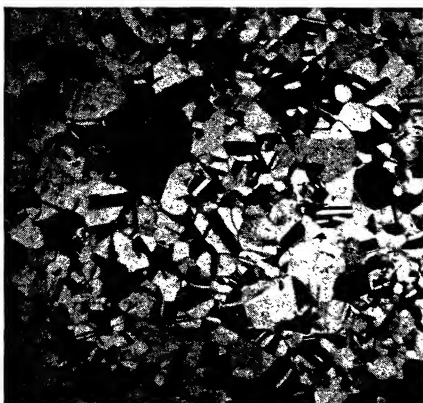


BRIN. 88.3—375° C.



BRIN. 82.6—400° C.

450° C.  
BRIN. 75,  $d = 0.020$  MM.500° C.  
BRIN. 70.4,  $d = 0.023$  MM.550° C.  
BRIN. 65.9,  $d = 0.033$  MM.FIG. 15.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .



600° C.  
BRIN. 61.7,  $d = 0.044$  MM.



650° C.  
BRIN. 55.8,  $d = 0.062$  MM.



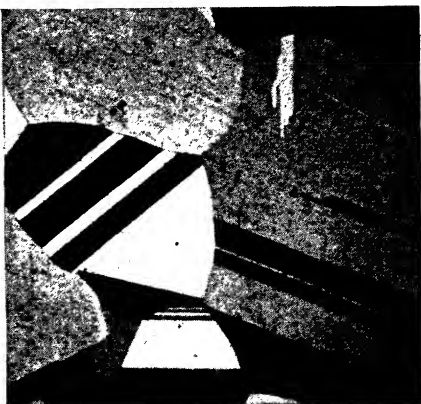
700° C.  
BRIN. 50.6,  $d = 0.110$  MM.



750° C.  
BRIN. 46.0,  $d = 0.197$  MM.



800° C.  
BRIN. 43.9,  $d = 0.300$  MM.



850° C.  
BRIN. 41.3,  $d = 0.448$  MM.

FIG. 16.—ETCHED WITH AMMONIA AND HYDROGEN PEROXIDE.  $\times 75$ .



rolled metal the drop in Brinell hardness indicates softening of the metal just before the new grains are seen; that the softening of lightly rolled metal progresses considerably before any new crystals can be detected; that the Brinell hardness of all specimens agrees closely from 600° C. to 850° C. As the previous anneal of the original hard specimens was about 650° C., the conclusion is drawn, and borne out by experience, that both the Brinell hardness and the grain size of annealed metal are greatly affected by the grain size due to the anneal previous to the anneal under discussion; and, as the grain size of the previous anneal diminishes, the Brinell hardness curves and the grain-size curves will approach those of metal annealed after very hard rolling as a limit; *i.e.*, in Fig. 1, the curve marked 59.1 per cent. reduction is this limit for the curves that represent more lightly rolled material. Figs. 14 to 16 illustrate the grain-size of 0.376-in. gage cartridge brass containing 69.20 per cent. copper, 30.76 per cent. zinc, 0.02 per cent. lead, and 0.02 per cent. iron. All specimens were reduced 35.1 per cent. before annealing.

For the direct comparison of the Brinell hardness with the diameter of the average grain in millimeters, a third plot (Fig. 3) has been added for the 0.130-in. (3.3-mm.) gage brass. This plot emphasizes two points brought out in the foregoing paragraph: (1) At low temperatures the grain size is influenced by the grain size of the previous anneal (0.060 mm.  $\pm$  in this case); and (2) from this grain size (0.060 mm.) upward, the Brinell hardness plotted against grain size gives a single curve no matter what the previous treatment of the metal has been. Consequently it may be repeated that in well-annealed brass of any given<sup>6</sup> alloy the Brinell hardness indicates grain size.

On account of the thickness of this particular brass as commercially used, the specimens were taken from a single bar as rolled in mill practice with a 35.1 per cent. reduction. In Fig. 2, the Brinell hardness and the grain size are plotted against annealing temperature so that they may be directly compared. The curves follow closely those of the 68 copper, 32 zinc alloy for the same percentage reduction and previous heat treatment.

### CONCLUSION

1. The grain sizes of the annealed alloys 68 copper, 32 zinc and 69 copper, 31 zinc agree closely when the previous heat treatment and reduction by rolling are made to correspond. The difference in thickness—0.374-in. (9.4-mm.) gage and 0.130-in. (3.3-mm.) gage—does not appreciably affect the grain size or the Brinell hardness.

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<sup>6</sup> The grain size and the Brinell hardness change progressively with the percentage of copper in brasses. The relation between the two, however, remains a constant for each brass mixture in the alpha phase.

2. The grain sizes of brasses annealed at low temperatures are greatly affected by the grain size and the reduction by rolling, previous to such annealing.

3. The grain size and Brinell data for the several conditions described, when plotted against temperature, give curves that approach the curve of metal annealed after hard rolling as a limit. It is desirable to select for standard of grain size (as determined by the temperature of annealing) those specimens that have been previously reduced by rolling at least 50 per cent.

4. In the case of cartridge brass of the composition 68 copper, 32 zinc, Brinell hardness indicates grain size. At low annealing temperatures the grain size is influenced by the grain size of the previous anneal. The finer the grain size of the previous anneal, the more closely will the curve, Brinell Hardness—Grain Size, approach the standard curve.

5. Since grain size is influenced by the grain size in the previous anneal and also by the amount of reduction by rolling previous to annealing, the hardness of cartridge brass may be determined with greater accuracy by the Brinell-hardness measurement than by attempting to judge it from the grain size.

## DISCUSSION

ARTHUR PHILLIPS,\* Bridgeport, Conn. (written discussion†).—It is to be regretted that the very valuable paper by Messrs. Bassett and Davis did not appera in the early war period. The data presented would have been of inestimable service to inspectors of cartridge brass who, admittedly, had little or no knowledge regarding the relation of grain size to temperature of anneal, and no real appreciation of the significance of the Brinell hardness test. The paper is of considerable interest to metallurgists also.

WALTER R. HIBBARD,‡ Bridgeport, Conn. (written discussion§) —The writer has carefully studied Messrs. Bassett and Davis's paper with considerable interest, inasmuch as our laboratory has tested cartridge brass by means of a Brinell machine. In March, 1917, at the suggestion of the Technical Department of the American Brass Co., we started a comparison of grain-size measurements and Brinell hardness of cartridge brass with a gage of 0.1 in. or greater. This was continued until March, 1918, when we adopted the Brinell hardness test as standard for brass 0.1 in. gage or greater, and discontinued grain-size measurements upon this metal. The data collected during the test showed that the Brinell hardness indicated more accurately how the metal acted in actual working operations. It was also more reliable because two manipulators check themselves closer by the Brinell test than by the grain-size measure-

\* Metallurgical Department, Bridgeport Brass Co.

† Received Jan. 18, 1919.

‡ The Remington Arms Union Metallic Cartridge Co.

§ Received Jan. 30, 1919.



ments. It also consumed less time in making the tests. Alfred V. de Forest, formerly assistant research engineer of our laboratories, has described the apparatus and some of the checking results in a paper read June, 1918, before the American Society for Testing Materials.<sup>1</sup> The writer hopes that a more satisfactory method for testing cartridge brass and gilding in gages thinner than 0.1 in. may be devised than the present method of grain-size measurements.

T. C. MERRIMAN, New Haven, Conn. (written discussion\*).—This most interesting paper gives much carefully obtained and valuable data. However, there are two points in connection with the commercial application of such data that might possibly be a source of trouble and misunderstanding. In the first place, the examination of thin sections of annealed brass, subjected to standard Brinell test (500 kg. on a 10-mm. diameter ball) at thicknesses from 0.075 to 0.150 in. (1.9 to 3.8 mm.) indicates that cold work has been performed on the specimen during the application of the load sufficient to extend way through the section and have the hardness of the metal affected by the backing. If the usual steel support is used, this effect is very evident. If a piece of soft brass is used as a support, there is an indentation in the surface of the support accompanied by a bulge on the under side of the tested specimen opposite the impression made by the 10-mm. ball. The result is that Brinell specifications (governmental or otherwise) on stock for a certain purpose may not be fair in all cases, since manufacturers, owing to differences in equipment, etc., may not all use precisely the thickness and conditions of stock on which the Brinell specifications were based. This would mean that the government Brinell specification might not give them the temper of stock best suited for their manufacturing methods.

The concluding paragraph of this paper, stating that "the hardness of cartridge brass may be determined with greater accuracy by the Brinell hardness measurement than by attempting to judge it from the grain size" appears to be in line with the movement of the last year or so among brass manufacturers toward substitution of the Brinell test for microscopic examination as an acceptance test on cartridge brass. The statement may be strictly true that the Brinell test is the best test for hardness, but it is not, as the statement might readily be construed, a sufficient test of suitable condition. For instance, some cartridge brass might accidentally have been overheated to a point where it would be distinctly unsafe to use for the manufacture of small arms cases and then be so rolled (reduction 3 to 5 per cent.) as to pass perfectly proper Brinell specifications. Under such conditions the microscope would reveal its unsuitability where the Brinell test had failed so to do.

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<sup>1</sup> *Proceedings*, American Society for Testing Materials (1918) **18**, Pt. 2, 449.

\*Received Feb. 24, 1919.

It is not intended to object to the Brinell test as a general test for the temper of cartridge brass, for I am in full agreement with the authors as to the usefulness and suitability of the Brinell test under many conditions, and I am sanguine that the development of the "Baby Brinell" will eliminate the likelihood of difficulty from the first point I have mentioned. However, Brinell specifications for cartridge brass are as yet given for 500-kg. load—10-mm. ball, and I firmly believe that even after the baby Brinell comes into fairly general use the Brinell test must be supplemented by frequent microscopic examinations and that microscopic inspection requirements should be retained as a vital part of cartridge-brass specifications.

C. H. MATHEWSON, New Haven, Conn. (written discussion\*).—Recent papers from Mr. Bassett's laboratory constitute a very welcome addition to the rather meager amount of scientific literature dealing with structure and properties, or in other words, the metallography of brass. They seem to have been developed mainly from the standpoint of supplying reliable and useful data that may be expected to further the intelligent handling of brass products. The collected data shown in the tables, when exhibited in graphic form, present several features of general interest and significance. There is a striking difference in the early parts of the several annealing curves shown in Fig. 1. While the heavily worked samples harden quite materially before they begin to soften, the lightly worked samples soften without any prior hardening.

The fact that hardening sometimes occurs after treatment at low temperatures before a true annealing effect begins has been known for some time, but, so far as I am aware, the supplementary information brought out by these curves is quite new. This early hardening has been attributed to a redistribution or relief of internal strain, but Howe considers this explanation hardly competent to account for similar effects of much greater intensity which occur in steel. Jeffries, however, in his discussion of the amorphous theory anticipates a condition of internal stress incident to the formation of amorphous metal,<sup>2</sup> which may be gradually relieved at ordinary temperature or more rapidly relieved at somewhat elevated temperatures. This explanation is quite in harmony with the observation that the more severe the initial deformation, the more pronounced the hardening in question.

We might even find a relation between season-cracking, an effect of internal strain, and this unique hardening. Thus, it is conceivable that metal which has not been worked severely enough to show an appreciable hardening when heated to about 200° C. will be stable under all conditions, while metal that hardens under this treatment will be subject to season cracking. This point appears to be worth some investigation.

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\* Received Feb. 17, 1919.

<sup>2</sup> This volume, p. 474.

It is noticeable that each curve of Fig. 1 intersects the curve lying below it in two localities before they merge into one common curve. The first intersection is due to a progressive lowering of the recrystallization temperature as the degree of deformation decreases and a reasonable explanation of these conditions has been given in the first paper cited by the authors. The second intersection indicates that when the more severely worked metal has nearly completed its recrystallization, and the less severely worked metal has recrystallized to a considerably lesser extent, both possess the same hardness. This would naturally occur at some characteristic temperature and the measured grain sizes would not be the same because there would be a greater number of grain areas composed of invisible fragments in the case of the less severely deformed material. These measurements would be greatly influenced by the grain size that existed prior to deformation and the authors have alluded to the bearing of this factor on the results.

It is quite probable that the less severely deformed material may develop abnormal grains, at favorable temperatures, by selective growth and this may account for the widening of the loops made by the last intersections of the curves of Fig. 1 as the degree of deformation decreases.

In the discussion of Dr. Jeffries' paper, I have referred to the relationship between Brinell hardness and grain size indicated by Fig. 3. of the paper by Messrs. Bassett and Davis. Using the equation:

$$\text{Brinell hardness} = K \frac{1}{\sqrt[3]{\text{diam. of average grain in mm.}}}$$

and placing the constant equal to 30 the following set of figures is obtained. These plot rather close to the curve shown in Fig. 3.

Brinell Hardness	Diameter of Average Grain, in Mm.
43	0.2401
50	0.1296
60	0.0625
75	0.0256
100	0.0081
150	0.0016

It is interesting to observe that beyond a hardness value of approximately 75, at which point grain-size measurements became impracticable, the authors carry dotted extensions of the curves up to the limiting hardness value of the cold-rolled metal, 160. In other words, they represent a continued decrease in grain size down to a minimum of zero size at the maximum hardness value.

Any attempt to count in this range would show a reversal of grain size with hardness and the grain size corresponding to maximum hardness

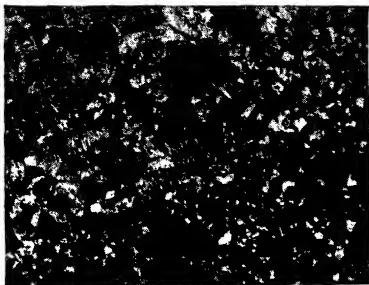


FIG. 1.

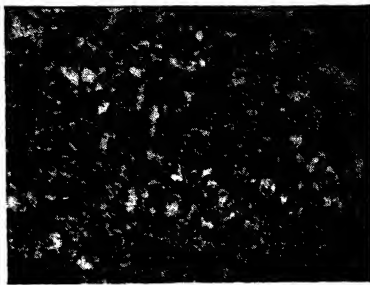


FIG. 2.

FIG. 1.—SHEET BRASS ROLLED 4 NUMBERS AND ANNEALED  $\frac{1}{2}$  HR. 350° C.  
 FIG. 2.—SHEET BRASS ROLLED 7 NUMBERS AND ANNEALED  $\frac{1}{2}$  HR. 350° C.

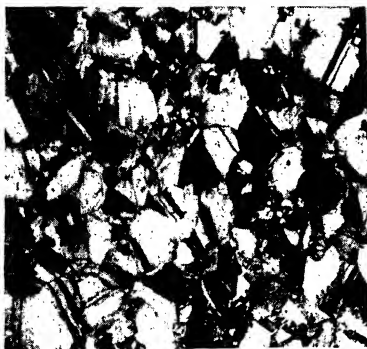


FIG. 3.



FIG. 4.

FIG. 3.—HIGH SIDE OF ECCENTRIC SHELL WHICH FAILED IN MERCURY TEST. 1 MIN. 800° F. (427° C.)

FIG. 4.—LOW SIDE OF ECCENTRIC SHELL WHICH FAILED IN MERCURY TEST. 1 MIN. 800° F. (427° C.)

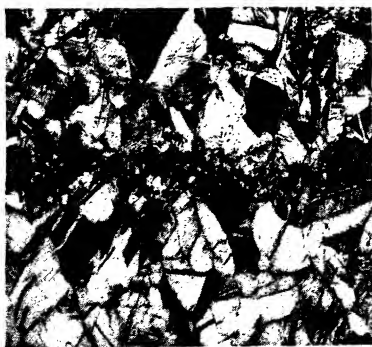


FIG. 5.

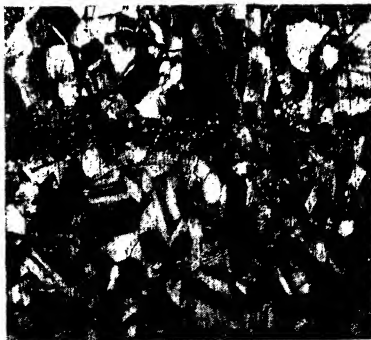


FIG. 6.

FIG. 5.—HIGH SIDE OF SCRATCHED ECCENTRIC SHELL. 1 MIN. 800° F. (427° C.)  
 FIG. 6.—LOW SIDE OF SCRATCHED ECCENTRIC SHELL. 1 MIN. 800° F. (427° C.)

$\text{NH}_4\text{OH} + \text{H}_2\text{O}_2 \times 75.$



FIG. 7.

FIG. 7.—HIGH SIDE OF SCRATCHED ECCENTRIC SHELL. 1 MIN. 850° F. (454° C.)

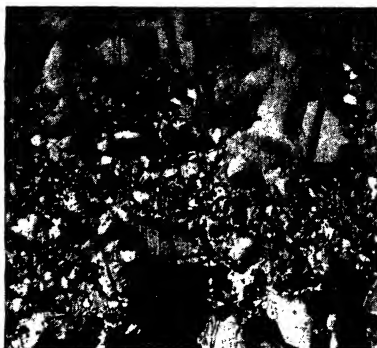


FIG. 8.

FIG. 8.—LOW SIDE OF SCRATCHED ECCENTRIC SHELL. 1 MIN. 850° F. (454° C.)

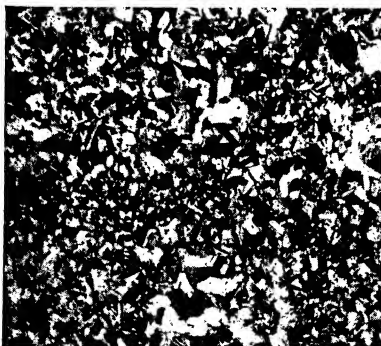


FIG. 9.

FIG. 9.—HIGH SIDE OF SCRATCHED ECCENTRIC SHELL. 1 MIN. 900° F. (482° C.)

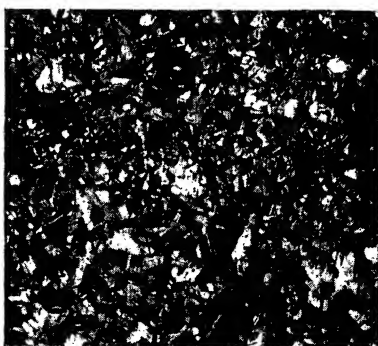


FIG. 10.

FIG. 10.—LOW SIDE OF SCRATCHED ECCENTRIC SHELL. 1 MIN. 900° F. (482° C.)

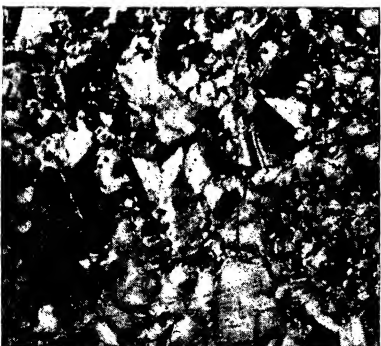


FIG. 11.

FIG. 11.—HALF SHELL 1 MIN. 800° F. (427° C.)

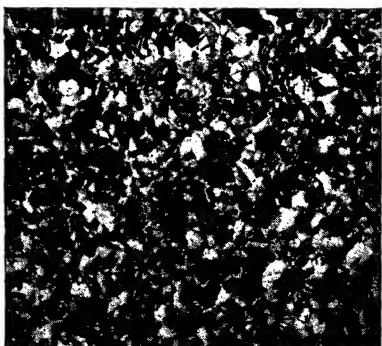


FIG. 12.

FIG. 12.—HALF SHELL 1 MIN. 900° F. (482° C.)

 $\text{NH}_4\text{OH} + \text{H}_2\text{O}_2 \times 75.$

would be the cold-worked equivalent of the original grain. The theoretical curve passes through decreasing values of grain size in this region down to a minimum of about 0.001 mm. at the maximum hardness value of about 160. This represents what I conceive to be the order of size of the indistinguishable crystalline grain fragments present in severely worked brass and I look upon the process of hardening by cold working as essentially a process of fragmentation with a building up of amorphous or subcrystalline boundaries.

W. B. PRICE,\* Waterbury, Conn. (written discussion†).—The practical application of Brinell hardness measurements in controlling the annealing of cartridge brass is of very great importance. While it is realized that the paper under discussion is supposed to deal only with the relation between grain size and Brinell hardness, it would have been interesting if other physical properties could have been added. In connection with the anneal shown at 350° C. with different reductions, analogous annealing experiments were carried out in this laboratory in 1914, with common high brass (copper, 64.13 per cent.; lead, 0.21 per cent.; iron, 0.04 per cent.; zinc, 35.62 per cent.) reduced four and seven numbers hard (Brown & Sharpe gage). These results are illustrated by Figs. 1 and 2.

It may be of interest to state in connection with the author's conclusions, "the grain size of brasses annealed at low temperatures are greatly affected by the grain size and the reduction by rolling previous to such annealing," an experience with low-temperature anneals on the mouth annealing of brass artillery cases. Occasionally a little trouble was experienced with a shell cracking on the mouth in the mercuric chloride test, and upon investigation it was found that failure usually took place upon the thin side of an eccentric shell. The photomicrographs of a shell that failed are shown in Figs. 3 and 4. The mouth anneal of 1 min. at 800° F. (427° C.) has caused a partial recrystallization on the thick, or more heavily worked, section, while there is very little evidence of recrystallization on the thin, or little worked, section. By raising the temperature of the anneal high enough, recrystallization was effected regardless of variation in amount of reduction on the mouth.

Another practical illustration of the important relationship between degree of cold working and temperature of recrystallization was noticed in a case that had been rather deeply scratched by an imperfect die previous to the mouth anneal. Examination under the microscope, after the annealing treatment, showed that recrystallization had taken place on the scratch but had not appeared on other parts of the case. Some cases were then purposely scratched and annealed for 1 min. at 800°, 850° and 900° F. (427°, 454° and 482° C.) (see Figs. 5 to 10). Recrystallization of the specimen was hardly effected at 850° F. (454° C.); incipient

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\* Scovill Mfg. Co.

† Received Feb. 17, 1919.

recrystallization has taken place both on the scratch and on some of the boundaries of the large deformed crystals. At 900° F. (482° C.), recrystallization has replaced the large originally deformed crystals with a much finer structure. The crystals on the scratch are much smaller than those on the unaltered section.

The relation of crystal size to tensile strength and per cent. of elongation is illustrated by the following experiment: A concentric shell was cut in two longitudinally; one half was annealed for 1 min. at 800° F. (427° C.) and the other half was annealed for 1 min. at 900° F. (482° C.). These are illustrated in Figs. 11 and 12. The 800° F. (427° C.) case when tested had a tensile strength of 57,385 lb. per sq. in. (4034 kg. per sq. cm.) and an elongation in 2 in. of 35.3 per cent., whereas the 900° F. (482° C.) case had a tensile strength of 51,567 lb. (3620 kg.) and an elongation of 52.2 per cent.

On pages 434 and 448, the authors state that in annealing hard rolled metal the drop in Brinell hardness indicates softening of the metal just before the new grains are seen. Upon examination of Fig. 1, page 433, however, showing graphically the relation between the Brinell hardness values and the annealing temperatures, it will be noted that the hardness curve rises perceptibly from the hard material to the 200° C. anneal for metal reduced 36.6 per cent. and upward. This indicates that for temperatures below visible recrystallization, possibly amorphous, material resulting from severe working undergoes some readjustment, which causes a distinct hardening instead of softening as would generally be expected. This phenomenon was observed by Mathewson and Phillips,<sup>3</sup> and came under my observation<sup>4</sup> when I annealed artillery cases at low temperatures.

J. BURNS READ,\* Washington, D. C.—There is nothing I can say other than in support of the data that Messrs. Bassett and Davis have furnished. In fairness to them, after what Mr. Phillips has said, I wish to state that these data were in the hands of officers of the Ordnance Department early last year and were very helpful in the manufacturing of cartridge cases for both small arms and artillery. Many contractors who undertook the manufacture of cartridge cases knew nothing of the control of brass quality through its working and anneal, consequently this information was most helpful and such troubles as too soft and too hard cases and season or corrosion cracking of cases were readily overcome through the application of the principles brought out in this article. A large number of micrographs and records of Brinell tests collected by the Ordnance Department certify as to the correctness of the data.

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\* Captain, Technical Staff, Ordnance Dept., U. S. A.

<sup>3</sup> *Trans.* (1916) **54**, 608-657.

<sup>4</sup> W. B. Price: American Society for Testing Materials (1918) **18**.

The question of Brinell tests has been given much attention by the Ordnance Department. As stated, the Brinell test has not been reliable when applied to thin material, and if it makes an impression through to the other side of the metal it is no longer a Brinell test.

The Ordnance Department in its laboratory at Pittsburgh has been working on the development of what it calls a baby Brinell machine, and has obtained some very satisfactory results in the brinelling of thin sheet metals. Since the signing of the armistice, this machine has been used in the direct brinelling of 0.30 caliber cartridge cases and very consistent results have been obtained. Because of these results, we feel that the brinelling of thin sheet metal is soon to be a reliable method of determining its hardness.

W. H. BASSETT.—The discussion of this paper has been gratifying and I am glad to hear the various conclusions that have been drawn from the data supplied. It was our intention to arrive at a practical method which would allow the rapid testing of cartridge brass in inspection. Of course, this work has a commercial bearing now that we are no longer manufacturing munition supplies. The Brinell standard test using the 10-mm. ball is not serviceable for thin metal, and I am glad to hear of the development of the baby Brinell; this should certainly offer a means for the more rapid testing of the hardness of thin brass.

Notwithstanding what Mr. Merriman said about the possibility of brass passing a Brinell hardness specification, in the case of over-annealing followed by light rolling, I do not believe that this fact detracts from the practicability of the Brinell test. It is, of course, possible by combinations of working to obtain false impressions from the grain count, as we attempted to bring out in the paper. If, for instance, work done on the brass is not sufficient to make apparent a deformation of the grain, the grain size will not be an accurate measure of the hardness of the material. In other words, the material may be much harder than the grain diameter indicates. Likewise, certain combinations may be arranged that will make the Brinell hardness figures misleading.

The purpose of proposing the Brinell hardness test to replace the grain count is to make possible more rapid inspection, but the two methods should be used in connection with each other. If the Brinell hardness test is used in inspection, an occasional microscopic examination should be made in order to obtain a proper understanding of the material being considered. So far as material above 0.100 in. thick is concerned, the Brinell hardness test can be trusted, provided an occasional microscopic examination is made.



## Babbitt and Babbitted Bearings

BY JESSE L. JONES,\* A. B., PITTSBURGH, PA.

(Milwaukee Meeting, October, 1918)

### SUMMARY

1. BRINELL tests at progressively increasing temperatures are given for a representative lead-base and a representative tin-base babbitt, showing that the former has superior resistance to deformation at the working temperatures of bearings.

2. Small squares of bearing bronze, tinned and then babbitted with a representative lead-base babbitt and a representative tin-base babbitt were subjected to compressive loading. The lead-base babbitt showed less average compression than the tin-base babbitt. Compression did not materially increase the Brinell hardness of the babbitts.

3. A process and a tool are described for giving smoother and more accurate surfaces to bearings than has heretofore been possible.

The following experiments have recently been carried out in the chemical laboratory of the Westinghouse Electric and Manufacturing Co., Pittsburgh, Pa. While the work is incomplete, it is described at the present time because data on the comparative merits of lead-base and tin-base babbitts are of interest in connection with the present shortage of tin.

### BRINELL HARDNESS OF BABBITT AT INCREASING TEMPERATURES

Bearings fail because of wiping, or deformation. Hence tenacity is desirable in a bearing metal, especially tenacity at high temperatures. The Brinell test is commonly regarded as a measure of tenacity. In fact, it has recently been proposed to substitute for Brinell hardness number the expression "tenacity number."<sup>1</sup> It seemed, therefore, that the Brinell test was especially adapted for the tests described.

Disks, 4 in. (101 mm.) in diameter and 1.5 in. (38 mm.) thick, were made of the babbitts to be studied, designated *A*, *B*, *C*. They were poured into metal molds, pyrometer leads being soldered in the center of each disk. The composition of the babbitts was as follows:

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\* Metallurgist, Westinghouse Elec. & Mfg. Co.

<sup>1</sup> J. W. Craggs: Notes on Testing Hardness of Metals. *Journal, Society of Chemical Industry* (1918) **37**, No. 3, 43T.

	A	B	C
Antimony.....	8	8½	14
Copper.....	2	8½	Nil
Lead.....	Nil	Nil	78
Tin.....	90	83½	8

The disks were heated by an electric hot plate, the heating being controlled by suitable rheostats. The thermocouple leads were connected with a Leeds & Northrup potentiometer, for measuring the temperatures. The disks were well insulated to prevent radiation losses, and after the desired temperature was reached it was held for several minutes to guard against variations.

The Brinell hardness tests were made on the bottom surfaces of the disks, a light machining cut being taken from each sample in order to

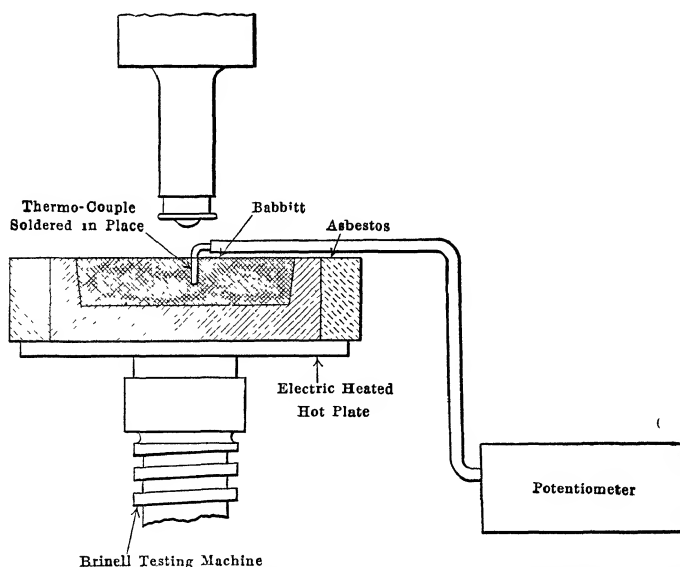


FIG. 1.—ARRANGEMENT OF APPARATUS FOR TESTING HARDNESS OF BABBITT AT VARYING TEMPERATURES.

secure a perfectly plane and smooth surface. Fig. 1 gives a sketch of the apparatus used for the test. The Brinell hardness numbers were plotted, giving the curves shown in Fig. 2.

At 35° C., the hardness of the A and C babbitts is identical, but above this temperature the lead-base babbitt has the greater hardness.

The curves for babbitts B and C are almost parallel, and not far apart; it is seen that the C curve is slowly approaching the B curve. Complete results from the various testing departments of the Westinghouse company, covering a number of years and a great variety of motors, confirm the superiority of the lead-base babbitt. The number of wiped bearings

that had to be rebabbitted was about 100 per month with the *A* babbitt, but not more than six per month when the *C* babbitt was used.

These practical results have led to the adoption of the *C* babbitt for all classes of machines, and the complete elimination of *A* or tin-base babbitt. Both experimentally and practically the lead-base babbitt has shown greater resistance to wiping or deformation at working temperatures than the tin-base babbitt.

The hard, genuine babbitt *B*, the test of which is shown in Fig. 2, while not regularly used, has been found very serviceable in the Kings-

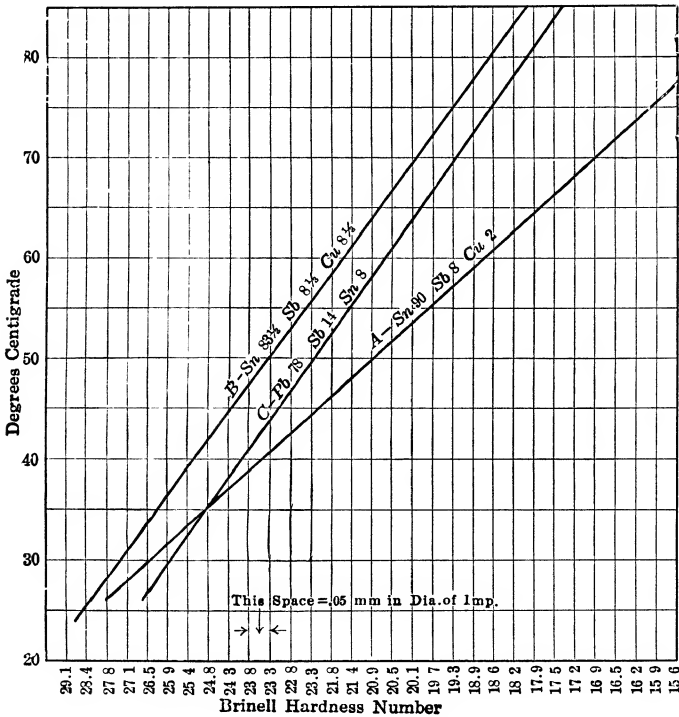


FIG. 2.—HARDNESS OF THREE BABBITS AT VARYING TEMPERATURES.

bury step bearing and other situations where the bearing pressures have been rather high. While the Brinell hardness shown in Fig. 2 for the *A* and *C* babbitts is not far from the average hardness found for these alloys when using the standard hardness test piece, the hardness observed for the *B* babbitt is much below normal; it should be about 38. The reason for the low Brinell hardness of this babbitt is probably the fact that it is difficult to prevent the large amount of copper in this alloy from segregating, even when it is kept very hot and stirred continuously. The *B* formula is almost the same as the genuine babbitt of the Society of Auto-

mobile Engineers. It is also similar to the alloy known as Fahrig metal, that is much used for lining aeroplane bearings abroad.

As it is the common belief among mechanical engineers that the addition of even a small amount of lead to a genuine babbitt renders it inferior, similar tests to those described above were run on the *A* babbitt, to which 1, 3 and 5 per cent. of lead had been added. The addition of 1 per cent. of lead to the *A* babbitt made a decided improvement in its resistance to deformation at increasing temperatures. Additions of more than 1 per cent. of lead did not increase the hardness in the same ratio.

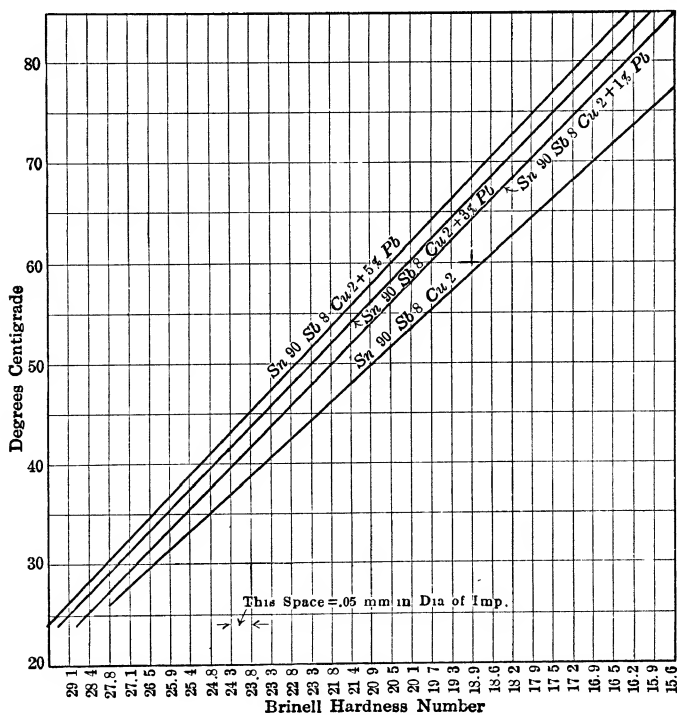


FIG. 3.—EFFECT OF LEAD ON HARDNESS OF *A* BABBITT.

The results of these tests are shown in Fig. 3, and are in harmony with the observations frequently made by users of babbitt, that when a small amount of lead has been added accidentally to a tin-base babbitt, its hardness and anti-frictional qualities have been much improved.

#### EFFECT OF COMPRESSION ON THE BRINELL HARDNESS OF BABBITS

After a babbitted bearing has been bored out nearly to size, it may next be scraped until an accurate fit is obtained. This is a tedious operation and demands a skilled mechanic; hence reaming or broaching are often used instead of this method. A reamer can be passed through a

number of bearings so that they will all be lined up in one operation. Reaming, at its best, however, is a rather violent operation, especially after the reamer becomes dull. Broaches are pushed through the bearing by a hydraulic press until the required dimensions are reached. In the case of large bearings, peening or compressing the babbitt by hammering is often specified. The last two operations, in particular, are supposed to compress the babbitt in a bearing and harden it so that it will give better service. The following experiment was made to ascertain the effect of compression of babbitt on its Brinell hardness.

Two phosphor-bronze plates (Cu, 80; Sn, 10; Pb, 10; P, 0.5 per cent.), 2 in. (50 mm.) square by  $\frac{1}{2}$  in. (12.7 mm.) thick, were machined all over;

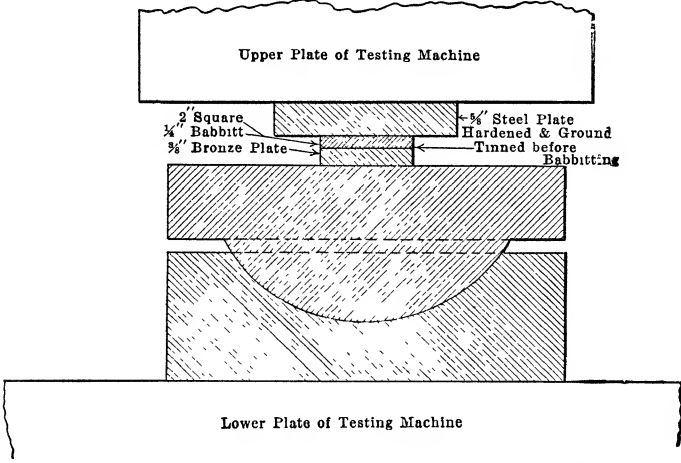


FIG. 4.—APPARATUS FOR COMPRESSIVE TESTS OF BABBITT.

one face was then tinned and babbitted with  $\frac{1}{4}$  in. (6.3 mm.) of babbitt, *B* babbitt being used on one sample and *C* babbitt on the other. The babbitt was machined to  $\frac{1}{4}$  in. and the samples subjected to a compressive test in the apparatus shown in Fig. 4, so designed that the load would be distributed as uniformly as possible. The successive loads and the corresponding Brinell hardness tests are given in Table 1.

TABLE 1.—*Compression and Hardness of Babbitts*

Load, lb. per sq. in.	<i>B</i> Babbitt		<i>C</i> Babbitt	
	Compression, in.	Brinell	Compression, in.	Brinell
8,500	0.0020	33.6	0.0015	23.8
10,000	0.0020	33.6	0.0020	23.8
11,000	0.0055	33.6	0.0030	24.8
12,000	0.0085	33.6	0.0032	24.8
13,000	0.0140	33.6	0.0102	24.8

The lead-base babbitt withstood compression better than the tin-base babbitt. When the load was increased to 30,000 lb. per square inch, however, the latter presented the better appearance, as the babbitt had flowed uniformly in all directions over the edge of the bronze square, while the lead-base babbitt compressed more on one side than on the other, and the sample tilted in spite of the rocker device. At a load of 30,000 lb. per square inch, the bronze also flowed appreciably.

These tests show that broaching, peening, etc., do not appreciably increase the hardness of babbitt, and that hardness must be obtained by quick cooling of the lining by water-cooled mandrels, etc.

Microscopic examination of a lead-base babbitt discloses a matrix or groundmass of several eutectics in which are embedded hard cubical

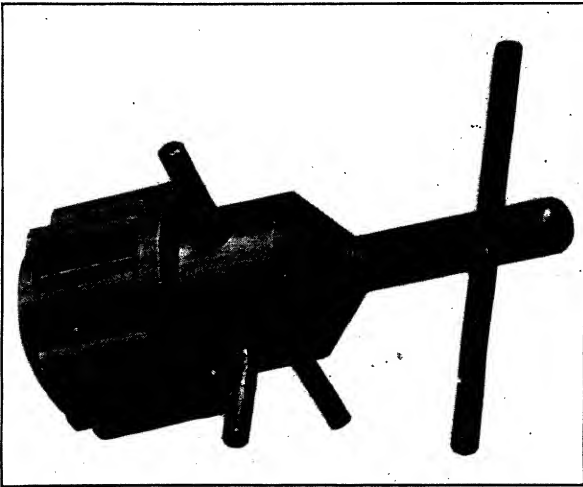


FIG. 5.—MILLS MICROMETER ROLLER.

crystals of tin antimonide. These crystals, being relatively low in specific gravity, tend to be more numerous in the upper portion of a babbitt lining. This lack of uniformity is guarded against by pouring a thin lining and chilling quickly. The secret of obtaining good bearings consists in keeping this matrix tough and hard. The same remark applies to the tin-base babbitts, although in these there is less tendency for the tin antimonide crystals to rise to the surface, owing to the lower specific gravity of these babbitts.

#### ROLLING OF BABBITTED LININGS BY THE MILLS MICROMETER ROLLER

As the above test of Brinell hardness of babbitt after compression showed practically no change in hardness, it was concluded that the various methods of finishing babbitted bearings, such as peening, broaching, and reaming, did not improve the hardness of the babbitt. As,

after these operations and the final scraping, it is still necessary to make a test run of bearings until the shaft is seated, it seemed possible to introduce improvement in the usual method of procedure. The so-called glass-like surface of old bearings is considered very desirable. Even if the babbitt is no harder than when cast, all inequalities of the surface have been smoothed out and friction has been reduced to a minimum. It occurred to the writer that, by a rolling or burnishing operation, this desirable finish could be given to babbitt linings so that a machine could be operated at full speed as soon as finished, without any trial run.

A tool, Fig. 5, designed by I. Mills, seems capable of doing this work satisfactorily. It was made for securing a better seat for the ball-bearing races of a small aeroplane wireless generator. A variation of 0.0007 in. (0.018 mm.) was found in the bearings, which could not be corrected by reaming or machining. With this tool, the seats in the aluminum alloy end brackets can be rolled to suit the individual variations of the ball bearings. A girl does the rolling, and the bearings can then be inserted or removed with the fingers. If the work is done by reaming, bearings may fit tightly at first, and after having been taken out may be quite loose, because burs, etc., have been removed.

While the Mills micrometer roller has been made only in a very small size as yet, it can be made in larger sizes and would then be capable of exerting much greater pressures. The present instrument is made for a diameter of  $1\frac{3}{4}$  in. (44.5 mm.) and has a range of 5 mils plus and minus. It consists of 10 rolls which can be expanded by a micrometer screw through the medium of two tapered cylinders which give a parallel outward movement to the rolls. The essential feature of the instrument is that the rolls are not equally spaced, as this would cause them to flute the rolled surface.

A trial of this method of rolling babbitted linings is being made on two sets of connecting-rod crank-shaft bearings of the Liberty engine, one set being filled with the lead-base babbitt *C*, and the other with the tin-base babbitt *B*. A description of the difficult conditions that are encountered in the bearings of the Liberty engine, due to its great power, are given in a very interesting paper, "The Metallurgist and the Aircraft Program" by Lieut. H. F. Wood, U. S. Army, Signal Corps.<sup>2</sup>

## DISCUSSION

G. H. CLAMER\* (Philadelphia, Pa.).—About 16,000 tons of tin is used annually in the production of white metals. The real reason for using the so-called genuine babbitt, which is a high-tin base babbitt,

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<sup>2</sup> *Proceedings*, Steel Treating Research Society (April, 1918) 1, No. 9, 15.

\* First Vice-president and Secretary, Ajax Metal Co.

perhaps in the majority of cases, is that tin has always been a high-priced metal and people naturally believe that what they pay a high price for must be good. Mr. Jones has shown that the common metal, lead, is a superior base for most purposes for which babbitt metal is used. The addition of lead is often looked upon as adulterant to genuine babbitt, and specifications for this reason rigidly restrict the lead content.

A sub-committee of the Society for Testing Materials, at the meeting in June, presented tentative specifications on babbitt metal, in which were included twelve formulas, containing from zero to 20 per cent. of tin, and several containing from 65 and 92 per cent. Babbitts containing from 21 to 64 per cent. of tin soften at slightly elevated temperatures, and so are not as good as the babbitt metals that are strictly of lead base and which carry but a small percentage of tin. However, these intermediate babbitts can be cast in very thin sections, owing to their low melting point. Experience has now proved that the very best kind of bearing is one having a bronze back and a very thin lining of babbitt, and the thinner the better. Babbitt metals with a fairly high melting point must be cast rather heavy and afterward machined out; there is a possibility that these intermediate babbitts might be cast thin enough to avoid the necessity for boring and then gotten down to a fine bearing surface by simply reaming or hobbing.

JESSE L. JONES.—It is not altogether a question of the desire to use a high-priced material, for its fluidity has a lot to do with the adaptability of a babbitt. Tin-base babbitts are quite fluid and you can cover a large area with a rather thin lining. The viscosity of a lead-base babbitt at the pouring temperature is such that it requires skill to pour a thin lining into a bearing that has very much of an area. But if a lead-base babbitt is highly refined, fluid, and properly poured by trained men, you can get a good solid lining, free from blowholes, and in every way equal to the tin-base babbitt lining. The necessary care and supervision are well repaid on account of the difference in price between the lead-base and the tin-base babbitt.

So far Dr. Frary's alloy of lead, hardened with barium and calcium, has not proved very practical. With bearings of considerable area, the alloy must be poured in sections of about  $\frac{1}{2}$  in. (12.7 mm.) in order to get it to run, and the oxidation loss is considerable. If the temperature is kept down to avoid the oxidation loss, there is lack of fluidity. Of course, by reducing the amount of barium and calcium, the fluidity is increased, but you do not get the hardness, and it is necessary to have the hardness also. The first samples submitted were of about 16.5 Brinnell hardness as cast, and hardened, in a few weeks, to about 19 or 20. We asked for material of higher Brinnell hardness but it proved too sluggish to run into thin linings.



## Metals and Alloys from a Colloid-chemical Viewpoint

BY JEROME ALEXANDER,\* M. SC., NEW YORK, N. Y.

(New York Meeting, February, 1919)

It is an outstanding fact of Nature that many of the practical properties of substances are dependent, not on their ultimate chemical composition, but on the kind and degree of aggregation of their constituent particles. Thus, a granite boulder is unmoved by wind or water, but if reduced to a fine dust it will be blown about by the wind and washed away by the rain. Carbon in one crystalline state of aggregation (diamond) is the hardest known substance, whereas in another crystalline state (graphite) it is so soft that it is used as a lubricant. Laying too much stress on the mere chemical analysis of substances is apt to obscure the fact that the nature of the aggregation of their particles is always a factor of importance, and sometimes the most important factor.

Colloid chemistry deals with matter in a very fine state of subdivision; its sphere begins with particles just a little smaller than a wave length of light and extends down until they blend into molecular dimensions. With particles of this size, such phenomena as surface tension and adsorption, which depend on the development of free surface, become enormously magnified. Thus a cube of 1 cm. edge has a surface of only 6 sq. cm.; but if it is reduced to colloidal dimensions by being cut up into cubes each having an edge of  $0.01\ \mu$  ( $0.00001\ \text{mm.}$ ), it will yield one million, million, million ( $1,000,000,000,000,000,000$ ) such cubes, having a combined area of 600 sq. m., or 21,274 sq. ft. The tiny force with which a drop of rain clings to the window pane becomes a factor to be reckoned with, if the surface involved is increased millions of times.

From the colloid-chemical viewpoint, metals and alloys may be regarded as jellies or sponge-like structures, the viscosity or stiffness of which at ordinary temperatures is exceedingly great; and like all jellies their properties are dependent on the composition and degree of dispersion of their constituent phases. These, in turn, depend on chemical composition, mutual solubility, speed of chilling, subsequent mechanical and heat treatment, etc. In fact, in preparing metals and alloys for practical use, we remove undesirable constituents (as in the conversion of pig iron into steel), add desirable constituents (as in

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\* Treasurer, National Gum & Mica Co.; Chairman, Special Committee on Colloids, Div. Chem. and Chem. Tech., National Research Council.

case-hardening or making alloy steels), or control the composition or particle size of the phases by chilling, rolling, or tempering.

It is a matter of importance which is the dispersed phase and which is the dispersing phase. For example, cream is an emulsion or dispersion of fat in water and wets paper, whereas butter is a dispersion of water in fat and greases paper. While I have not yet examined the experimental facts in the case of metals, it seems probable that the relative surface tensions of the phases toward each other are important factors in determining the constitution of the matrix or dispersion medium, and in determining which substances shall constitute the dispersed phases. Substances that lower surface tension tend to collect at the interfaces and produce in the matrix, as well as in the dispersed phases, a fine degree of subdivision. Thus, according to Putz, the predominant effect of vanadium in steel is to decrease the size of the ferrite grains and make the material harder; it renders the ordinary structure due to pearlite fine-grained and homogeneous.

I believe that the application of the ultramicroscope to the study of the minute structure of metals will reveal much that is of interest. The great difficulty is in preparing sections of sufficient tenuity for examination. One means of preparing such thin metallic films was devised by Faraday, who floated gold leaf upon dilute cyanide solutions; and an intimation of what results may be obtained was given by Sir George Beilby in his Hurter Memorial Lecture entitled *The Surface Structure of Solids*.<sup>1</sup>

Colloids exert a powerful influence on crystallization. Thus plaster of Paris crystallized from pure water shows characteristic long interlacing crystals. The addition to the water of even 0.01 per cent. of gelatine delays the set and practically inhibits the crystallization, there being formed instead spherocrystals. With the addition of 0.5 per cent. of gelatine, the time of set was increased from 40 min. to 960 min. Most metals have inherently a powerful tendency to crystallize, which in many cases is strongly inhibited by various substances present in the melt; and since even small quantities may produce the result, it is obvious that they are in a very finely dispersed state, and in many cases, probably colloidal.

The change in dispersion of a substance with varying chemical composition of the dispersion medium may be illustrated by an experiment with ordinary soap, which dissolves in alcohol into a clear crystalloidally dispersed solution; even in the ultramicroscope no particles are visible. In water, however, soap forms a cloudy colloidal solution; and if sufficient water is added to the alcoholic solution, a turbidity at once becomes manifest. If water is added to the slide under the

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<sup>1</sup> *Journal, Society of Chemical Industry* (1903) **22**, 1166.

ultramicroscope, the alcoholic solution literally explodes into millions of actively moving ultramicros of colloidal dimensions. Another strong analogy to metals is exhibited by transparent soap. When quickly chilled, it shows very small ultramicros, whereas when slowly cooled the ultramicros are much larger and may make the soap cloudy. Still another analogy is found in gold ruby glass. When quickly cooled, this is colorless and shows no ultramicros; but when reheated, the invisible nuclei of metallic gold grow into visibility in the ultramicroscope and the glass develops a color. If the colorless glass has been formed under conditions insuring the formation of a very large number of nuclei, the glass when reheated develops a large number of small ultramicros, and the color is a rich ruby red. If the initial chilling has been too slow, the nuclei are too few and too large, and when the glass is reheated there are formed a relatively small number of large masses of metallic gold, which give the glass a violet or dirty blue color. In such spoiled ruby glass, the aggregations of gold may grow until they are visible to the naked eye.

From what has just been said we can easily understand that when metals are heated so as to reduce their viscosity sufficiently, their particles move to establish the tendencies toward aggregation that were arrested by the high viscosity consequent upon solidification. This accounts for most of the phenomena consequent upon tempering or annealing. Even at ordinary temperatures gold and lead diffuse into each other, but the motion of their particles is so slow that it is quite invisible in the microscope and can be determined only after a long lapse of time. In fact, a cork, if given sufficient time, will gradually float to the top of a barrel of tar or asphalt.

Finally we might mention the diffusion of gases into and through metals. The adsorption of gas is primarily a surface phenomenon. Particles within the mass of a phase are on all sides surrounded by particles of the same kind. Particles at the surface, however, have one side in contact with a different phase. The free surface or interface may thus become the seat of a residual attraction or surface affinity. This is exhibited by freshly cleaved sheets of mica, which adhere if promptly put together, but in a few moments this property is lost owing to the adsorption of atmospheric gases. If there is a diminution of attraction at an interface, fracture will preferentially follow such interface, as is evident in coarsely crystalline metals, for example.

In the case of finely dispersed metallic systems, with enormous free interfaces, the adsorption of gas may weaken the attraction between the particles and render the metal brittle. In the case of gases with actively moving particles, the metal may be actually penetrated by the gas, as those who have to handle hydrogen know to their sorrow.

In conclusion, I must emphasize the fact that in Nature all transitions are gradual. Ordinary coarse suspensions pass imperceptibly into and through the colloidal zone to actual crystalloidal subdivision, or so-called "true solution." Sols pass so gradually into gels that we cannot point out the exact spot where one ends and the other begins; and there is no sharp line of demarcation between physics and chemistry, which are connected by a twilight zone of colloidal phenomena partaking of the nature of both.

## DISCUSSION

JEROME ALEXANDER.—All of you undoubtedly know that a microscopic examination of metals will reveal many things; but we should go further than just simply using a low- or medium-powered microscope and seek the reason why grain size varies under different conditions. When we get below the limits of the ordinary microscope, we enter the so-called colloidal field, which means that we are dealing with aggregations of matter that approximate, say, 100 millimicrons. The colloidal dimensions average approximately 50 millionths of a millimeter and extend down to about 3  $\mu\mu$ . This, of course, is beyond the range of visibility in the ordinary microscope. Just to give you an idea of this matter, if 1 cu. in. of platinum is reduced to a sheet five molecules thick, it will cover 7 acres; you can imagine the amount of surface involved when metal, or anything else, is subdivided to so high a degree. Of course five molecules thick is a little below the limits of the colloidal dimensions.

The statement is made in the paper that "metals and alloys might be regarded as jellies and sponge-like structures." Of course that is not absolutely true, because, at certain stages, metals are rather aggregations of crystals surrounded by some kind of a matrix. But when metals or alloys are chilled suddenly there is not the degree of crystallization ordinarily found in the ordinary commercial metals.

It is very difficult to make examinations in masses of metals. About 9 or 10 years ago, I did some work showing the effect of colloidal substances, such as glue, etc., on the crystallization of ordinary salts, like sodium chloride, etc. If ordinary sodium chloride is mixed with a small per cent. of gum arabic and allowed to crystallize, the most interesting branching forms are obtained, which at once reminded me of a great many of the so-called crystal forms seen in the photomicrographs of alloys. It appears that wherever there is a crystallization tendency it is possible, by the presence of other substances, to modify that tendency so that the final result is something between what the metal or the salt would have done had the influencing substances been absent, and some other extreme, which has not yet been determined. In any event, crystallization is very powerfully influenced by other substances. Now, whether the

action is physical or chemical, or whatever you wish to call it, the fact of the matter is that wherever an ordinary salt, like sodium chloride, starts to crystallize, you can influence its whole crystallization by adding to it a little gum arabic or similar colloid.

It seemed to me that a similar condition must be true in metals. I have not delved into the whole question of metals; I know very little about them, but the analogies are so strong that it seems a very promising field of investigation, and the only difficult question is the preparation of the proper samples of investigation so as to go further than just using the ordinary microscope on the metals. We should see whether we cannot get some information by means of an instrument that will render visible particles smaller than are shown by the ordinary microscope. You might raise the question, are these particles crystalline particles or not? My answer to that would be that I do not believe we can ever know with absolute certainty. Since they are smaller than a wave length of light, they never will have any apparent size or shape; they simply will vary in brilliance and in apparent size. Consequently, the particle itself can never be resolved, although it may be made visible. There is a big difference between visibility and resolvability. Besides, substances like glue and gum arabic have the power not only of altering crystallization but of preventing crystallization. There must be an analogous condition in the case of metals and, from simply glancing through the metallurgical literature, I have seen numerous cases which point to that very fact.

As an illustration, I have some samples of transparent soap that were prepared about 9 or 10 years ago. Transparent soap has, in a way, some peculiarities that offer a strong analogy to metals. A piece of this soap was melted in a pot over the kitchen stove and one part was cooled relatively slowly; the other part was cooled off quickly. When examined with the ultra microscope, the one that had been chilled very quickly showed practically no ultramicros; it was, practically speaking, homogeneous. The piece that had been slowly cooled showed a large number of ultramicros. When examined about 3 or 4 years later, no ultramicros were visible in the quickly cooled piece, which was apparently as homogeneous as before, but in the slowly cooled pieces there were crystals visible under low power. There is also a big difference between the two pieces of soap even today.

WILDER D. BANCROFT,\* Washington, D. C. (written discussion†).—In two-phase systems there are three possibilities and Mr. Alexander has only considered two. We may have the first phase the internal

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\* Lieut.-Colonel, Chemical Warfare Service, U. S. A., Acting Chief, Research Division.

† Received Jan. 31, 1919.

one and the second the external one; we may have the first phase the external one and the second phase the internal one; and we may have both phases continuous. This last case occurs when we have a roll of wire fencing standing in the air; the wire is continuous and so is the air. It is quite probable that this case occurs very often in alloys and it is, therefore, important that it should not be overlooked. With gold containing a little bismuth, the bismuth forms a coating around the grains of gold and consequently makes a brittle alloy having a low conductivity; this is one of the cases covered by Mr. Alexander's classification. The important nature of the external phase is shown very clearly in some work by C. G. Fink published some years ago. He took the same mixtures of metallic tungsten and alumina and compressed them to solid masses, the only difference being that the tungsten was relatively coarse in one case and relatively fine in the other. When the tungsten was relatively coarse, the fine alumina particles coated the coarser tungsten particles and he obtained a white mass that does not conduct electricity. When the tungsten particles were relatively fine, they coated the coarser alumina particles and he got a black mass that conducts electricity.

It seems a pity to bring up an analogy between alloys and jellies, because the alloy is the simple case and the jelly is the complicated one. In the case of the alloy the mass is always crystalline; we can determine easily the composition of the two phases and it is not difficult to ascertain whether we are dealing with external and internal phases, or what I call an interlaced system. In the case of a jelly, both phases are amorphous; we do not know the composition of either phase and we do not know the structure. It may be advisable some day to discuss jellies with reference to alloys, but it is certainly a step backward to discuss alloys with reference to jellies.

PAUL D. MERICA,\* Washington, D. C. (written discussion†).—The study of the effect of degree of dispersion of solid solutions in alloys seems to hold forth most interesting possibilities. Until recently only one example of an alloy solution of varying dispersion of the solute was well known to metallurgists; that of cementite ( $\text{Fe}_3\text{C}$ ) in alpha, or perhaps in alpha plus beta, iron. According to one theory of the hardening of steel, the changes taking place during the tempering of steel are due to the progressive coalescence of this cementite from particles of almost atomic size, in martensite, to colloidal particles of larger size in troostite and sorbite, and finally to particles of size sufficiently large to be visible under the microscope, in granular pearlite. As the average size of particle of cementite increases, the hardness of the conglomerate diminishes and the electrical conductivity increases.

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\* U. S. Bureau of Standards.

† Received Feb. 26, 1919.

I have recently studied another alloy series in which also a solid solution occurs, apparently in different degrees of dispersion. This is the binary system: copper-aluminum, in which a solution of approximately 4 per cent. of copper as  $\text{CuAl}_2$  in solid aluminum is formed at about  $500^\circ\text{C}$ . The solubility of the  $\text{CuAl}_2$  in aluminum diminishes with decreasing temperature; consequently such an alloy, when quenched from  $500^\circ\text{C}$ ., is supersaturated with respect to  $\text{CuAl}_2$ . During the tempering of the quenched alloy at about  $200^\circ\text{C}$ ., as all evidence seems to indicate, the excess particles of  $\text{CuAl}_2$  coalesce just as do those of cementite, forming progressively larger and larger ones.

Just after quenching, when the solution is still presumably in atomic dispersion, it is quite soft, as the coalescence progresses during tempering, the hardness first increases to a maximum and then diminishes again. There is apparently a certain degree of dispersion which produces maximum hardness in this solution, one in which the particles are greater than when in atomic dispersion.

It is to be hoped that the introduction of the use of the ultra-violet microscope and of the  $x$ -ray diffraction "microscope" for the study of the structure of metals and alloys may make possible further systematic study of the effect of degree of dispersion upon the physical properties of solid solutions.

ZAY JEFFRIES,\* Cleveland, Ohio (written discussion†).—The microscope has, indeed, proved a mighty tool in the study of the structures of substances. It is limited in its resolving power to the wave length of the light used for illumination. A little in resolving power can be gained by using ultraviolet rays, which can only be detected by the photographic plate. Even the highest powered microscope only permits the study of aggregates of millions of atoms. The  $x$ -ray spectrometer, on the other hand, makes it possible to study the positions of the atoms in a crystal. The positions of the atoms in over fifty crystals have now been ascertained. But in amorphous substances the  $x$ -ray spectrometer is only able to tell us that the atoms seem to have no regular arrangement; the actual arrangement and method of grouping is still a mystery.

In most perfectly formed crystals and in so-called "grains" in metals, the  $x$ -ray spectrometer teaches us that colloids seem to play no role inasmuch as the atoms are arranged in regular layers rather than in random clusters. In the boundary region between crystalline grains of like as well as unlike substances and in amorphous substances like glass, it seems probable that the random clusters, or colloids, play an important role.

There still remains the application of wave lengths connecting the ultraviolet and  $x$ -rays both by direct reflection or diffraction and by the

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\* Director of Research, Aluminum Castings Co.

† Received Mar. 24, 1919.

ultramicroscope. In the study of substances like glass, for example, it may be possible to identify the clusters or molecules by one wave length and to study the positions of the atoms in a cluster by using a smaller wave length. Any light that can be thrown on this important unknown subject and its relation to the properties of substances is therefore very welcome, and Mr. Alexander's paper is so written as to stimulate thought and experimentation in this direction.

JEROME ALEXANDER (author's reply to discussion\*).—Col. Bancroft states that in the case of alloys, the mass is always crystalline. While this generally may be so in the case of alloys, as ordinarily met with, before the alloy reaches this state or condition, its constituent particles must be in a still more finely dispersed state. In alloys, the speed of transition between the finely dispersed state where the phases are amorphous, and the crystalline state where the phases are of known composition, is so great that, in most cases, the amorphous condition is not met with in the final product. My point is that by quick chilling or the introduction of substances that retard or interfere with crystallization, the final degree of dispersion may be powerfully influenced, and by such influence the properties of the resulting metal may also be affected. It is perfectly proper, therefore, to consider the analogy to the initial amorphous, or jelly, state as well as to the final, or crystalline, state and I have done both. In the case of transparent soap, it was possible to show both the amorphous and the crystalline states in the same substance.

Moissan observed that if a little platinum be added to mercury the mercury will then readily emulsify with water. I mention this as an instance of the effect of one metal on the surface tension or aggregation tendency of another. I also refer to my work on the effect of colloids on crystallization,<sup>2</sup> as showing the powerful effect of such substances upon crystallization.

Of course, as Col. Bancroft points out, where both phases of a two-phase system are continuous, such condition must be considered; but even in this case the degree of dispersion of the phases is an important factor. I believe that more knowledge may be acquired by applying the ultramicroscope and other methods of colloid research to metals and alloys, and even though most metals and alloys ultimately become crystalline, that we should investigate their dispersed, or amorphous, state, and follow so far as possible the changes that finally lead to the crystalline condition and the means by which this condition may be modified or prevented.

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\* Received Mar. 17, 1919.



## Effect of Temperature, Deformation, and Grain Size on the Mechanical Properties of Metals\*

BY ZAY JEFFRIES,† MET. E., D. SC., CLEVELAND, O.

(New York Meeting, February, 1919)

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### INTRODUCTION

DURING the period 1914 to 1916, the writer observed many times that tungsten wire could be drawn hot until it became brittle but on cooling to room temperature it became ductile again. In searching the literature for parallel cases, it was found that Beilby<sup>1</sup> had produced hard-drawn wires of gold, silver, and copper that showed less than 1 per cent.

\* Part 2 of thesis submitted to Harvard University for degree of Doctor of Science, May 1, 1918. Part 1 appeared in *Journal*, Institute of Metals (No. 2, 1918).

† Director of Research, Aluminum Castings Co.

<sup>1</sup> G. T. Beilby: The Hard and Soft States in Metals. *Journal*, Institute of Metals (No. 2, 1911) 6, 5-43.

elongation at room temperature but when tested at the temperature of liquid air showed elongations as high as 12 per cent. At the same time the tenacity was greater at the lower temperature. Beilby offered no explanation for this, but he assumed that a general increase in tenacity favored ductility. That this is only incidental and not the real cause of the ductility is shown by Hadfield's<sup>2</sup> experiments on iron at liquid-air temperatures, which show an even greater increase in tenacity than Beilby's results but in which the ductility was reduced to substantially zero.

To find, if possible, an explanation of these phenomena and to study other allied phenomena investigations were undertaken, the results of some of which were reported in another paper.<sup>3</sup>

#### NATURE OF EXPERIMENTS

Experiments were carried out on tungsten, iron, and copper. Tungsten has the highest melting point of all the metals and the highest recrystallization temperature and its behavior at room temperature is exactly opposite to that of the common ductile metals as regards the properties of malleability and ductility. When it is given a treatment that anneals other metals, it is made brittle, and when worked severely below its recrystallization temperature and then cooled to room temperature, it is ductile. A study of the properties of tungsten at low temperatures should therefore throw light on the properties of metals in general at temperatures relatively low for them. For example, tungsten might be considered to behave at room temperature the same as copper at some lower temperature, which the results of these experiments indicate must be lower than the temperature of liquid air.

Iron was selected because it has the peculiar property of being about 25 per cent. stronger at 200°–250° C. than at room temperature. It also has intermediate melting and recrystallization temperatures between those of tungsten and copper. Copper should be representative of the soft ductile metals, such as gold, silver, platinum, and lead.

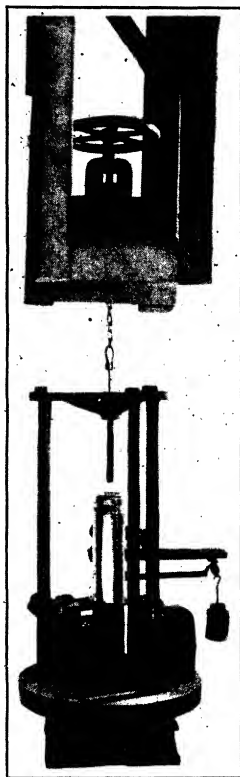


FIG. 1.

<sup>2</sup> R. A. Hadfield: Experiments Relating to the Effect on Mechanical and Other Properties of Iron and Its Alloys Produced by Liquid Air Temperatures. *Journal, Iron and Steel Institute* (No. 1, 1905) 67, 147–219.

<sup>3</sup> Zay Jeffries: *Metallography of Tungsten*. This volume.

## MATERIALS USED IN EXPERIMENTS

The copper was electrolytic bus-bar stock and showed by analysis about 99.9 per cent. copper. Microscopic examination showed that it contained about the same percentage of copper oxide as is ordinarily found in wire-bar copper.

Tungsten used commercially for incandescent filaments containing about 0.75 per cent. thoria was selected for these experiments. Many microscopic and chemical analyses of this material show that the metallic part is practically pure tungsten and that thoria is present in small non-metallic spherical globules.

The iron was kindly furnished by the American Rolling Mill Co. of Middletown, Ohio. It is the variety known as "Armco" iron and contained about 0.02 per cent. carbon. It was furnished in bars 0.25 in. by 1 in. (6.35 by 25.4 mm.).

## DESCRIPTION OF SAMPLES

C-1.—Copper drawn at room temperature from 0.125 in. to 0.025 in. (3.175 mm. to 0.635 mm.) without intermediate annealing. The bus-bar was swaged to 0.125 in. and then annealed and the annealed piece was drawn cold without further annealing to the finished size. This is 96 per cent. reduction. Fig. 4 is a micrograph  $\times 150$ .

C-2.—Same as C-1, annealed 5 min. at 750° C. Fig. 6 is a micrograph  $\times 150$ .

C-3.—Copper drawn at room temperature from 0.042 in. to 0.025 in. (1.0668 mm. to 0.635 mm.) without intermediate annealing; 64 per cent. reduction. Fig. 7 is a micrograph  $\times 150$ .

C-4.—Copper drawn at room temperature from 0.031 in. to 0.025 in. (0.7874 mm. to 0.635 mm.) without intermediate annealing; 64 per cent. reduction. Fig. 10 is a micrograph  $\times 150$ .

C-5.—Copper drawn at 150° C. from 0.075 in. to 0.025 in. (1.905 mm. to 0.635 mm.) without intermediate annealing; 90 per cent. reduction. Fig. 12 is a micrograph  $\times 150$ .

C-6.—Copper drawn at 150° C. from 0.042 in. to 0.025 in. (1.0668 mm. to 0.635 mm.) without intermediate annealing; 64 per cent. reduction. Fig. 14 is a micrograph  $\times 150$ .

C-7.—Copper drawn at 150° C. from 0.031 in. to 0.025 in. (0.7874 mm. to 0.635 mm.) without intermediate annealing; 32 per cent. reduction. Fig. 16 is a micrograph  $\times 150$ .

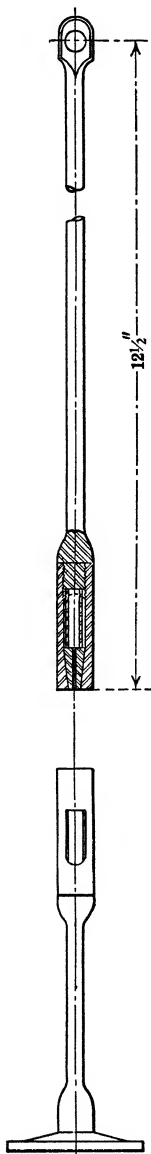


FIG. 2.

*C*-8.—The same as *C*-1, heated for 10 min. at 150° C. in oil.

*C*-9.—Copper drawn at 200° C. from 0.031 in. to 0.025 in. (0.7874 mm. to 0.635 mm.) diameter without intermediate annealing; 32 per cent. reduction. Fig. 18 is a micrograph  $\times 150$ .

*C*-10.—The same as *C*-1 heated 3 min. at 450° C. Fig. 22 is a micrograph  $\times 150$ .

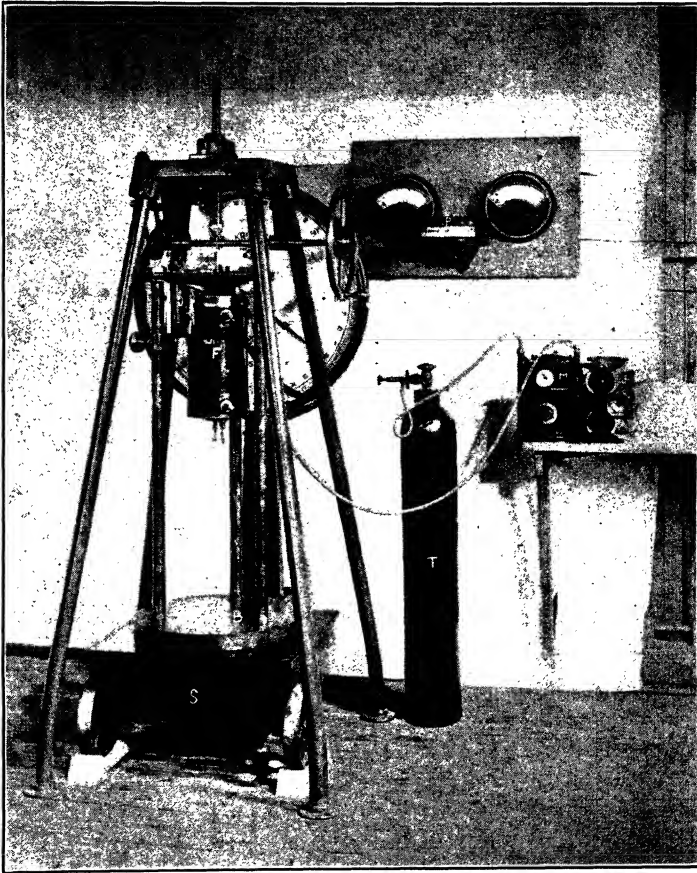


FIG. 3.—*S*, SCALE; *B*, BASE PORTION; *F*, ELECTRIC FURNACE; *J*, COLD JUNCTION (THERMOS BOTTLE); *C*, CLAMP; *L*, LOADING MECHANISM; *P*, GALVANOMETER FOR TEMPERATURE MEASUREMENT; *T*, TANK OF ARGON; *M*, METER TO MEASURE FLOW OF ARGON; *R*, RHEOSTAT TO CONTROL TEMPERATURE OF FURNACE.

*T*-1.—Tungsten wire 0.025 in. (0.635 mm.) diameter produced by swaging a rod 0.165 in. (4.191 mm.) to 0.030 in. (0.762 mm.) diameter and drawing from 0.030 in. to 0.025 in. At the beginning of swaging a temperature of about 1300° C. was used and this was gradually decreased as the working progressed, the finishing temperature being 1000° C. This

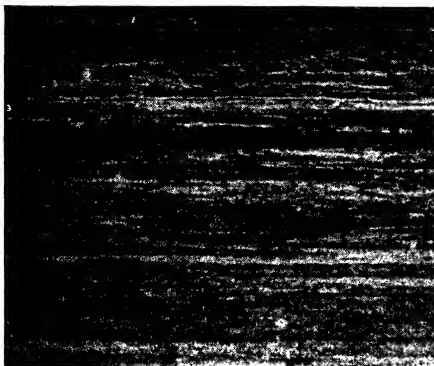


FIG. 4.—C-1. LONGITUDINAL SECTION OF COPPER WIRE DRAWN AT ROOM TEMPERATURE FROM 0.125 IN. TO 0.025 IN.  $\times 150$ .



FIG. 5.—C-1-c-950. SAME AS FIG. 4 AFTER TENSILE TEST AT 950° C.  $\times 150$ .



FIG. 6.—C-2. COPPER ANNEALED 5 MIN. AT 750° C. LONGITUDINAL SECTION.  $\times 150$ .

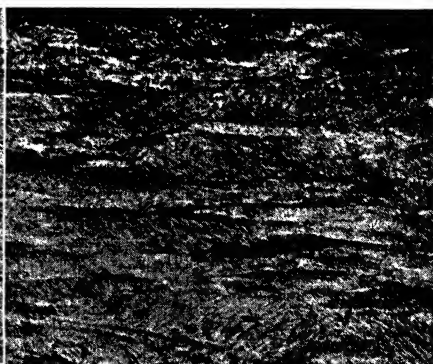


FIG. 7.—C-3. COPPER DRAWN AT ROOM TEMPERATURE FROM 0.042 IN. TO 0.025 IN. LONGITUDINAL SECTION.  $\times 150$ .



FIG. 8.—C-3-a-750. SAME AS FIG. 7, BROKEN IN 5 SEC. AT 750° C.  $\times 150$ .



FIG. 9.—C-3-c-750. SAME, BROKEN IN 3 MIN. AT 750° C.  $\times 150$ .

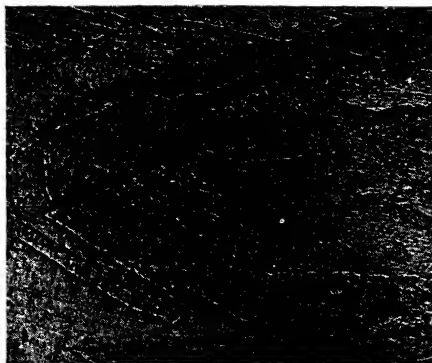


FIG. 10.—C-4. COPPER DRAWN AT ROOM TEMPERATURE FROM 0.031 IN. TO 0.025 IN.  $\times 150$ .

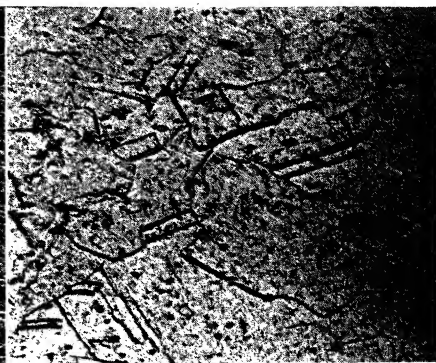


FIG. 11.—C-4-a-750. SAME AS FIG. 10, AFTER TENSILE TEST AT 750° C.  $\times 150$ .



FIG. 12.—C-5. COPPER DRAWN AT 150° C. FROM 0.075 IN. TO 0.025 IN.  $\times 150$ .

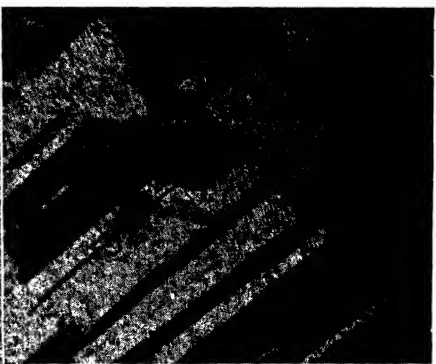


FIG. 13.—C-5-a-750. SAME AS FIG. 12, AFTER TENSILE TEST AT 750° C.  $\times 150$ .

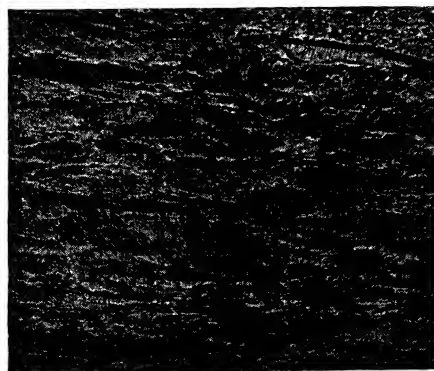


FIG. 14.—C-6. COPPER DRAWN AT 150° C. FROM 0.042 IN. TO 0.025 IN.  $\times 150$ .

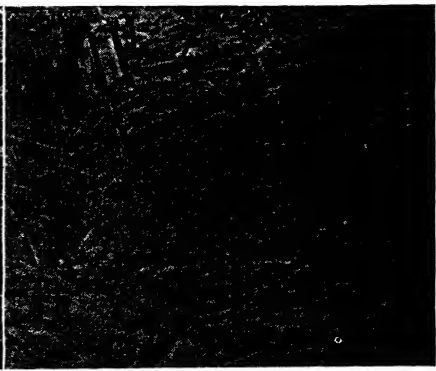


FIG. 15.—C-6-a-750. SAME AS FIG. 14, AFTER TENSILE TEST AT 750° C.  $\times 150$ .



FIG. 16.—C-7. COPPER DRAWN AT 150° C. FROM 0.031 IN. TO 0.025 IN.  $\times 150$ .

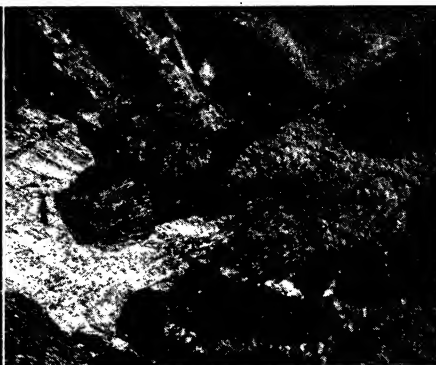


FIG. 17.—C-8-a-750. COPPER DRAWN FROM 0.125 IN. TO 0.025 IN. COLD, HEATED 10 MIN. AT 150° C. AND THEN TESTED AT 750° C.  $\times 150$ .



FIG. 18.—C-9. COPPER DRAWN AT 200° C. FROM 0.031 IN. TO 0.025 IN.  $\times 150$ .



FIG. 19.—C-9-a-750. SAME AS FIG. 18, AFTER TENSILE TEST AT 750° C.  $\times 150$ .

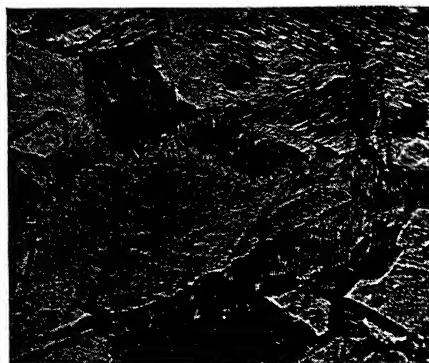


FIG. 20.—C-9-a-950. COPPER DRAWN AT 200° C. FROM 0.031 IN. TO 0.025 IN. AND TESTED AT 950° C. BROKEN IN 3 MIN.  $\times 150$ .

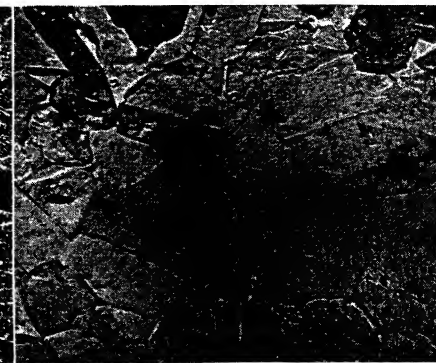


FIG. 21.—C-9-b-950. SAME AS FIG. 20 BUT BROKEN IN 5 SEC.  $\times 150$ .

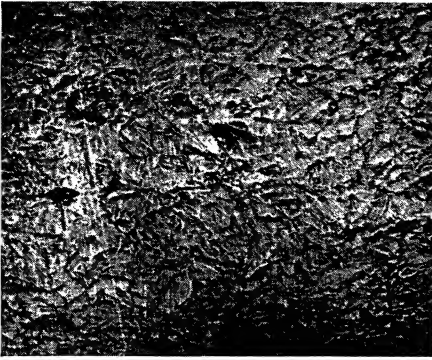


FIG. 22.—C-10. COPPER ANNEALED  
AT 450° C. FOR 3 MIN.  $\times 150$ .

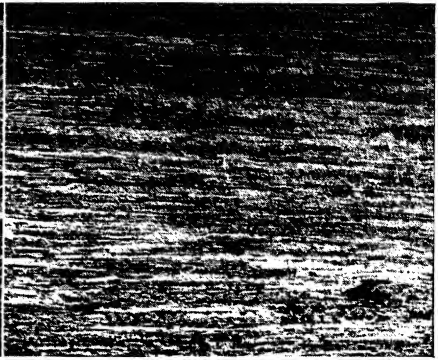


FIG. 23.—T-1. TUNGSTEN SWAGED  
AND DRAWN HOT FROM 0.165 IN. TO  
0.025 IN.  $\times 150$ .

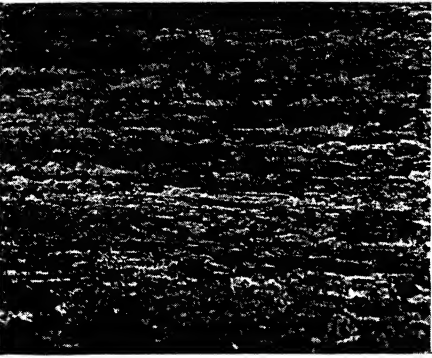


FIG. 24.—T-2. SAME AS FIG. 23 BUT  
EQUIAXED.  $\times 150$ .

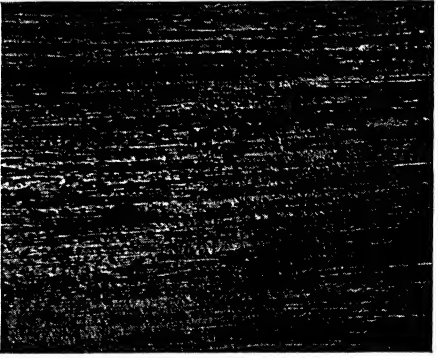


FIG. 25.—T-3. TUNGSTEN SWAGED  
AND DRAWN AT 1000° C. FROM 0.125 IN.  
TO 0.025 IN.  $\times 150$ .

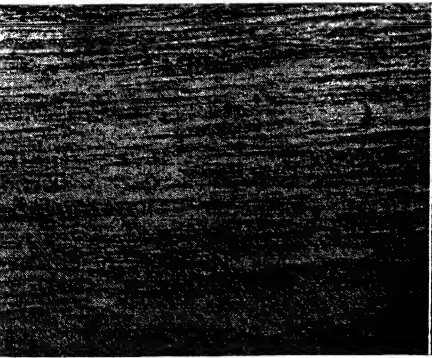


FIG. 26.—T-4. TUNGSTEN EQUIAXED  
AT 0.042 IN. AND THEN DRAWN AT 1000°  
C. TO 0.028 IN.  $\times 150$ .



FIG. 27.—I-1. ARMCO IRON DRAWN COLD  
FROM 0.120 IN. TO 0.025 IN.  $\times 150$ .



is about 97.5 per cent. reduction by working below the recrystallization temperature without annealing. Fig. 23 is a micrograph  $\times 150$ .

*T-2.*—The same as *T-1* equiaxed by heating to about 2000° C. with electric current in an atmosphere of hydrogen. Fig. 24 is a micrograph  $\times 150$ .

*T-3.*—Tungsten equiaxed at 0.125 in. (3.175 mm.) diameter and then swaged at 1000° C. to 0.030 in. (0.762 mm.) and drawn to 0.025 in. (0.635 mm.). This is 96 per cent. reduction by working below the recrystallization temperature. Fig. 25 is a micrograph  $\times 150$ .

*T-4.*—Tungsten equiaxed at 0.042 in. (1.0668 mm.) diameter and drawn at 1000° C. to 0.028 in. (0.7112 mm.). This is 56 per cent. reduction by drawing below the recrystallization temperature. Fig. 26 is a micrograph  $\times 150$ .

*I-1.*—Armco iron drawn at room temperature from 0.120 in. to 0.025 in. (3.048 mm. to 0.635 mm.) diameter. The 0.25-in. (6.35-mm.). Armco iron bar was swaged to 0.120 in. and then annealed and the annealed wire was drawn without further annealing to the final size. This represents about 96 per cent. reduction of area by cold drawing without annealing. Fig. 27 is a micrograph  $\times 150$ .

*I-2.*—Same as *I-1*, annealed 5 min. at 850° C. Fig. 32 is a micrograph  $\times 150$ .

*I-3.*—Armco iron drawn at room temperature from 0.042 in. to 0.025 in. (1.0668 mm. to 0.635 mm.). Some of the Armco iron was annealed at 0.042 in. and then drawn cold without intermediate annealing to 0.025 in. This represents 64 per cent. reduction in area by cold drawing without annealing.

*I-4.*—Armco iron drawn at room temperature from 0.031 in. to 0.025 in. (0.7874 mm. to 0.635 mm.) diameter. Some iron was annealed at 0.031 in. diameter and drawn without further annealing to 0.025 in. This represents 32 per cent. reduction of area by cold drawing. Fig. 33 is a micrograph  $\times 150$ .

*I-5.*—Armco iron drawn at 400° C. from 0.075 in. to 0.025 in. (1.905 mm. to 0.635 mm.) without intermediate annealing. This represents a reduction of about 90 per cent. without annealing. Fig. 34 is a micrograph  $\times 150$ .

*I-6.*—Armco iron drawn at 400° C. from 0.042 in. to 0.025 in. (1.0668 mm. to 0.635 mm.) without intermediate annealing. This is 64 per cent. reduction. Fig. 36 is a micrograph  $\times 150$ .

*I-7.*—Armco iron drawn at 400° C. from 0.031 in. diameter to 0.025 in. (0.7874 mm. to 0.635 mm.) diameter without intermediate annealing. This is 32 per cent. reduction. Fig. 37 is a micrograph  $\times 150$ .

*I-8.*—The same as *I-1*, heated for 10 min. at 400° C. in lead bath.

*I-9.*—Armco iron drawn at 275° C. from 0.042 in. to 0.025 in. (1.0668 mm. to 0.635 mm.) without intermediate annealing. This is 64 per cent. reduction. Fig. 39 is a micrograph  $\times 150$ .



FIG. 28.—I-1-c-575. SAME AS FIG. 27 AFTER TENSILE TEST AT 575° C.  $\times 150$ .



FIG. 29.—I-1-a-690. ARMCO IRON (I-1) AFTER TENSILE TEST AT 690° C.  $\times 150$ .



FIG. 30.—I-1-a-850. ARMCO IRON (I-1) AFTER TENSILE TEST AT 850° C.  $\times 150$ .



FIG. 31.—I-1-c-950. ARMCO IRON (I-1) AFTER TENSILE TEST AT 950° C.  $\times 150$ .

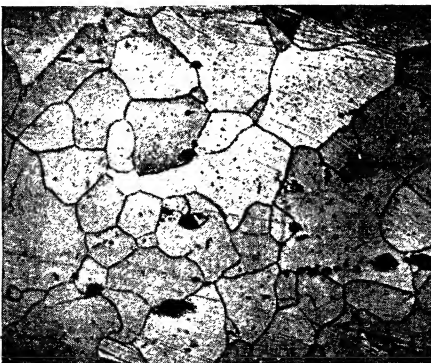


FIG. 32.—I-2. ARMCO IRON WIRE 0.025 IN. DIAMETER. ANNEALED 5 MIN. AT 850° C.  $\times 150$ .



FIG. 33 —I-4. ARMCO IRON DRAWN COLD FROM 0.031 IN. TO 0.025 IN.  $\times 150$ .



FIG. 34.—I-5. ARMCO IRON DRAWN AT 400° C. FROM 0.075 IN. TO 0.025 IN.  $\times 150$ .



FIG. 35.—I-5-c-575. ARMCO IRON (I-5) AFTER TENSILE TEST AT 575° C.  $\times 150$ .



FIG. 36.—I-6. ARMCO IRON DRAWN AT 400° C. FROM 0.042 IN. TO 0.025 IN.  $\times 150$ .

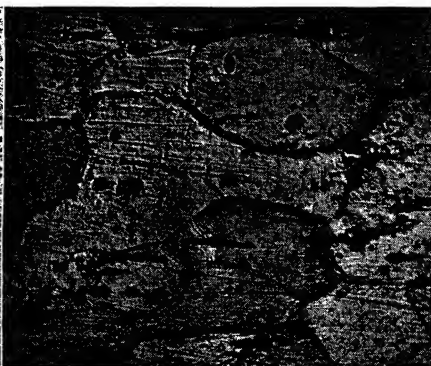


FIG. 37.—I-7. ARMCO IRON DRAWN AT 400° C. FROM 0.031 IN. TO 0.025 IN.  $\times 150$ .

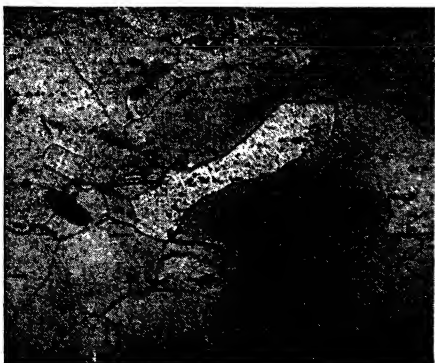


FIG. 38.—I-7-b-575. ARMCO IRON (I-7) AFTER TENSILE TEST AT 575° C.  $\times 150$ .

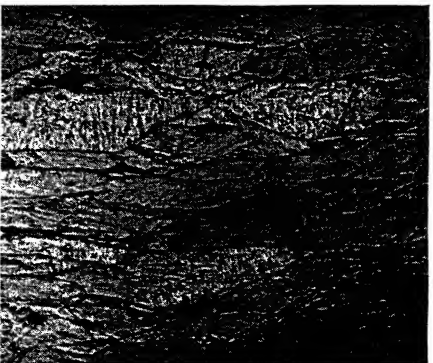


FIG. 39.—I-9. ARMCO IRON DRAWN AT 275° C. FROM 0.042 IN. TO 0.025 IN.  $\times 150$ .

*I-10*.—Armco iron drawn at 275° C. from 0.031 in. to 0.025 in. (0.7874 mm. to 0.635 mm.) without intermediate annealing. Fig. 40 is a micrograph  $\times 150$ .

*I-11*.—The same as *I-1*, heated 3 min. at 600° C. Fig. 41 is a micrograph  $\times 150$

*I-12*.—Same as *I-1* annealed for 15 min. at 750° C. in electric furnace. Fig. 42 is a micrograph  $\times 150$ .



FIG. 40.—*I-10*. ARMCO IRON DRAWN AT 275° C. FROM 0.031 IN. TO 0.025 IN.  $\times 150$ .



FIG. 41.—*I-11*. ARMCO IRON (*I-1*) AFTER HEATING 3 MIN. AT 600° C.  $\times 150$ .



FIG. 42.—*I-12*. ARMCO IRON (*I-1*) ANNEALED 15 MIN. AT 750° C.  $\times 150$ .

The hot drawing of iron at 400° C. was done with the help of a lead bath maintained at the proper temperature. The die was not immersed but was just outside the molten lead. The hot drawing of copper and of the iron at 275° C. was done in an oil bath. The dies were immersed in the oil. The temperature of the tungsten during drawing was maintained by means of a suitable gas burner. The temperature was determined from the color of the wire with the help of an optical pyrometer.

## DETAILS OF TESTS AND APPARATUS

The wires were subjected to tensile tests at temperatures from  $-190^{\circ}\text{C}$ . to a maximum of  $900^{\circ}$ – $1000^{\circ}\text{C}$ . No attempt was made to determine the elastic limit but the other tensile properties, namely, tensile strength, elongation, and reduction of area, were determined at all temperatures.

The apparatus used for these tests, shown in Fig. 1, is composed of two main parts, the base and the loading mechanism. The base consists of two cast-iron disks, a bottom and top that are fastened together by three pieces of steel pipe, as shown. The base weighs a little over 100 lb. (45 kg.) and is placed on a platform scale. The top of the base portion is provided with a steel tube 0.875 in. (22.225 mm.) outside and 0.5 in. (12.7 mm.) inside diameter, inside of which the test pieces are clamped by the special clamps shown in Fig. 2. The upper clamp is connected to the loading mechanism. Additional weights can be set on the lower part of the base if necessary. When the load is applied by the hand wheel, shown in Fig. 1, the test wire pulls up on the base and the amount of its pull is measured on the weighing mechanism of the scale. The zero point on the scale is equal to the weight of the base; the scale is kept balanced continually during the test until the wire breaks. The scale reading at the breaking load of the test specimen is then subtracted from the zero reading and the difference gives the breaking load of the test specimen.

This scale arrangement was suggested by Prof. H. W. Hayward, of Massachusetts Institute of Technology. A general idea of the type and size of apparatus needed for these tests was given by the writer to L. S. Twomey of the Cleveland Wire Division of the General Electric Co., who designed the base and clamps.

*Liquid-air Tests.*—Punch marks 2 in. (50.8 mm.) apart were made on all wires and the clamps were set about 2.5 in. apart, leaving about 0.25 in. between each clamp and the closest punch mark. The test wires were locked in the clamps and inserted in position in the machine and a 1-qt. wide-mouthed thermos bottle of the food-jar type, more than three-quarters filled with liquid air, was raised in such a manner that the steel tube containing the test wire was immersed in the liquid air. The test wire was completely immersed and hence its temperature was that of boiling liquid air. The vacuum jar and platform to hold it in position during test are shown in Fig. 1.

As soon as violent boiling ceased, load was applied until the test wire broke. The vacuum jar containing the liquid air was then lowered from the steel tube and another vessel containing warm water was substituted for it. When the temperature had been raised by the water the clamps were removed. The elongation of the wire was measured in the usual manner.

*Room-temperature Tests.*—The room-temperature tests were made

with the same apparatus without any bath to maintain constant temperature.

*Tests at 100° C.*—These tests were made in boiling water. An electric percolator heater was placed on the platform instead of the vacuum jar and a coffee pot was used to hold the water. The test specimen was set the same as for the liquid-air experiment but was immersed in boiling water. The water was kept boiling vigorously from the beginning to the end of any given test.

*Tests at 200° C.*—These tests were made in hot crisco. It was found that crisco could be heated to 250° C. with very little volatilization; one of the chief advantages of crisco is that it is very fluid at the higher temperatures. The crisco was heated with the same electric heater that was used for the water and in the same coffee pot. The temperature during any given test may have varied as much as 3° or 4°. Several pounds of crisco, however, were used in the coffee pot, so that temperature changes were slow. Immediately after each test piece broke, the temperature of the crisco was measured with a mercury thermometer and recorded.

*Tests Above 200° C.*—These tests were made at the laboratories of the Cleveland Wire Division of the General Electric Co. A different scale and different loading mechanism were used and the base of the apparatus was modified so that an electric furnace could be used to obtain the proper temperature. This apparatus is shown in Fig. 3.

A Kron scale with a 30-in. (76.2-cm.) dial graduated in quarter pounds but sensitive to less was used. The electric furnace consisted of a 1 in. (25.4 mm.), inside diameter, alundum tube 12 in. long wound with nichrome ribbon enclosed in a gas-tight steel cylinder. Powdered silica was used as a heat-insulating medium between the alundum tube and the steel cylinder. The gas-tight steel cylinder was provided with a connection to a tank of compressed argon, so that a neutral atmosphere could be maintained in the furnace at the higher temperatures. Argon was selected because hydrogen reacts with the copper oxide in copper and modifies its properties at high temperatures and it did not seem advisable to risk the effect of nitrogen on iron at these temperatures. Argon is known to be inert to all three metals used.

The electric furnace was so fastened that the load on the wires was transmitted to the base portion by the lower end of the furnace. The bottom clamp was flanged to fit a seat at the bottom of the furnace housing; the flange was so large that it could not have been drawn through the furnace. The upper clamp was small and could go easily through the furnace tube. This clamp was fastened to the loading mechanism by means of a clevice, as shown in Fig. 3. As in the other arrangement the zero point was the weight of the base of the apparatus (including the furnace) and the load required to break the test wire was obtained by

subtracting the scale reading at the maximum load of the test wire from the scale reading before load had been applied.

A Pt.-pt.rh. thermocouple was used to measure the temperature. The hot junction was placed in the furnace tube about half way between the two clamps, that is, at about the central portion of the test wire. The thermocouple was connected with a Wilson-Maeulen galvanometer with both millivolt and temperature scales. The temperature of the electric furnace could be maintained constant with the help of a wire-wound rheostat. An ammeter and voltmeter were in the circuit, as shown in Fig. 3. The loading mechanism, clamps, and electric furnace were designed by L. S. Twomey.

In testing a wire, it was first fastened to both top and bottom clamps. The top clamp was then fastened to a wire and pulled through the furnace tube from the bottom and fastened to the loading mechanism. A hole in the top of the base, large enough for the top clamp to pass through, also provided a slight clearance for the escape of the argon from the electric furnace. When the flange on the bottom clamp engaged its seat, additional upward motion of the loading mechanism stressed the test wire. There was a cover for the bottom of the furnace tube, which could be made gas-tight by means of a gasket joint. When the wire was broken during test, the bottom clamp dropped a fraction of an inch and rested on this cover. The test wire could easily be removed from the furnace without bending.

#### DETERMINATION OF ELONGATION

The elongation was determined in the usual manner by making punch marks on the wires 2 in. (50.8 mm.) apart and measuring the distance between punch marks after the wires had been broken. There were comparatively few instances of fractures in the punch marks. Where these at times occurred, the results obtained were about the same as in the same wires with center breaks. Occasionally the marks were made so deep that the wires were weakened at the punch marks; these tests were either thrown out or are indicated. It was also observed that when the break occurred outside the marks the per cent. elongation was but slightly different from that with a center break. This would be expected because the diameter of the wires is so small when compared to the distance between punch marks (about 1 to 80) that a marked reduction of area would have but little influence on the elongation.

#### DETERMINATION OF REDUCTION OF AREA

The final diameters, at the point of break were determined with a microscope and a micrometer eyepiece. After trying several conditions, it was found that best results were obtained with a magnification of about 25 diameters using direct sunlight for illumination. The reduction of area measurements are subject to an error of about 2 per cent.

## RESULTS OF TESTS

The results of the tests are given in Tables 1 to 25, inclusive, and the graphs are shown in Figs. 43 to 73, inclusive.

TABLE 1.—*Mechanical Properties at Various Temperatures of Copper Wire (C-1) Drawn Cold Without Intermediate Annealing from 0.125 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-1-a-L	-190	80,600	7.00	60 0	These samples tested in liquid air.
C-1-b-L	-190	80,600	5.50	51 5	
Average..	....	80,600	6.25	55 7	
C-1-a-R	Room	67,400	0.75	69 0	
C-1-b-R	Room	67,400	0 75	60 0	
Average..	....	67,400	0 75	64 5	
C-1-a-100	100	61,000	0 75	65 0	Tested in boiling water.
C-1-b-100	100	62,500	0 75	69 0	
Average..	....	61,750	0.75	67.0	
C-1-a-200	215	45,000	0 00	69 0	Tested in hot crisco.
C-1-b-200	215	45,000	0 35	72 5	
Average..	....	45,000	0.17	70 7	
C-1-a-330	330	22,400	14.00	88 0	Partly annealed during test. Load applied rapidly to avoid annealing. Tested in electric furnace.
C-1-b-330	330	36,500	1.55	80 0	
C-1-a-450	450	10,700	31 25	41 0	Broken in about 1 min. Fast loading.
C-1-b-450	450	9,700	25 00	30 0	Broken in about 10 min. Slow loading.
C-1-a-600	600	8,700	28 00	95 6	15 sec. to break. Fast loading.
C-1-b-600	600	5,100	20 00	18 0	5 min. to break. Slow loading.
C-1-a-750	750	4,600	20 00	99 5	10 sec. to break. Fast loading.
C-1-b-750	750	2,500	4.70	5 0	15 min. to break. Slow loading.
C-1-a-950	950	2,500	17 00	98 0	5 sec. to break.
C-1-b-950	950	710	4 70	5 0	1 min. to break.
C-1-c-950	950	1,000	4 70	11 0	2 min. to break.



TABLE 2.—*Mechanical Properties at Various Temperatures of Copper Wire (C-2) Drawn Cold from 0.125 to 0.025 In. and then Annealed at 750° C.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent	Reduction of Area, Per Cent	Remarks
C-2-a-L	-190	46,900	27.50	78 0	Tested in liquid air.
C-2-b-L	-190	44,800	24 50	65 0	
Average..	. . . .	45,850	26 00	72.0	
C-2-a-R	Room	30,600	24 00	76 0	
C-2-b-R	Room	31,700	25 00	76.0	
Average..	. . . . .	31,150	24 50	76 0	
C-2-a-100	100	27,500	27 50	84 0	Tested in boiling water.
C-2-b-100	100	28,500	22 50	74 0	
C-2-c-100	100	28,500	22 50	74 0	
Average..	.	28,170	24.10	77.0	
C-2-a-200	206	22,000	17 00	81 0	Tested in hot crisco.
C-2-b-200	215	23,200	20 25	78 0	
Average.	. . .	22,600	18 70	79 5	
C-2-a-330	330	20,500	18 75	48.5	Tested in electric furnace.
C-2-b-330	330	20,500	17 10	47.0	
Average..	. . . . .	20,500	17.90	47.7	
C-2-a-450	450	9,700	13.50	24.0	Loaded slowly.
C-2-b-450	450	10,700	11.00	47.0	Loaded slowly.
C-2-c-450	450	14,700	22.00	47.0	Broken in 10 sec.
C-2-d-450	450	12,700	17.00	24.0	Broken in 2 min.
C-2-a-600	600	8,700	16.50	15.0	Broken in 15 sec.
C-2-b-600	600	6,100	14.00	13.0	Broken in 3 min.
C-2-a-750	750	4,600	5.50	75 0	Broken in mark in 10 sec.
C-2-b-750	750	3,100	9.00	10 0	Broken in mark in 3 min.
C-2-a-950	950	710	4.70	5.0	Broken in 30 sec.
C-2-b-950	950	710	3.10	3.0	Broken in 2 min.
C-10-a-R*	Room	38,000	35 00	82.0	
C-10-b-R	Room	36,000	35 00		

\* Annealed at lower temperature and hence smaller grain size.

TABLE 3.—*Mechanical Properties at Various Temperatures of Copper Wire (C-3) Drawn Cold from 0.042 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-3-a-L	-190	77,600	8.25	58.0	Tested in liquid air.
C-3-b-L	-190	77,600	8.25	60.0	
Average..	.....	77,600	8.25	59.0	
C-3-a-R	Room	59,200	1.50	65.0	
C-3-b-R	Room	59,200	2.30	67.0	
Average..	.....	59,200	1.90	66.0	
C-3-a-100	100	55,000	0.75	69.0	Tested in boiling water.
C-3-b-100	100	54,000	0.75	65.0	
Average..	....	54,500	0.75	67.0	
C-3-a-200	218	45,000	0.75	76.0	Tested in hot crisco.
C-3-b-200	214	43,000	1.15	76.0	
Average..	.....	44,000	0.95	76.0	
C-3-a-330	330	41,000	1.55	76.0	Tested in electric furnace.
C-3-b-330	330	40,000	1.55	76.0	
Average..	.....	40,500	1.55	76.0	
C-3-a-450	450	11,200	17.75	30.0	Loaded rapidly.
C-3-b-450	465	10,200	14.90	18.0	Loaded slowly.
C-3-a-600	600	7,650	25.00	51.5	Broken in 10 sec.
C-3-b-600	600	7,150	12.50	24.0	Broken in 2 min.
C-3-a-750	750	4,600	23.50	99.5	Broken in 5 sec.
C-3-b-750	750	3,100	5.50	24.0	Broken in 2 min. Broke in mark.
C-3-c-750	750	3,100	12.50	15.0	Broken in 3 min. Broke in center.

The interpretation of the results given in Tables 1 to 25, inclusive, has for its basis the amorphous theory in strain-hardened metals as developed by Beilby<sup>4</sup> and in annealed metals as advocated by Bengough<sup>5</sup> and Rosen-

<sup>4</sup> *Op. cit.*

<sup>5</sup> G. D. Bengough: A Study of the Properties of Alloys at High Temperatures. *Journal, Institute of Metals* (No. 1, 1912) 7, 123-174.

TABLE 4.—*Mechanical Properties at Various Temperatures of Copper Wire (C-4) Drawn Cold from 0.031 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-4-a-L	-190	66,400	7.85	69.0	Tested in liquid air.
C-4-b-L	-190	69,400	9.00	60 0	
Average..	.....	67,900	8.40	64.5	
C-4-a-R	Room	47,000	2.30	65 0	
C-4-b-R	Room	45,900	1.95	67 0	Tested in boiling water.
Average..	.....	46,450	2.12	66 0	
C-4-a-100	100	45,000	2.35	60 0	
C-4-b-100	100	45,000	1.55	72.5	
Average..	.....	45,000	1.90	66.2	Tested in hot crisco.
C-4-a-200	217	37,000	0.75	65.0	
C-4-b-200	213	37,000	0.75	72 5	
Average..	.....	37,000	0 75	68.7	
C-4-a-330	320	33,500	1.55	76.0	Tested in electric furnace.
C-4-b-330	320	33,500	1.55	76.0	
Average..	.....	33,500	1.55	76 0	
C-4-a-450	450	12,200	22.00	41.0	Loaded rapidly.
C-4-b-450	450	9,700	11.80	30.0	Loaded slowly.
C-4-a-600	600	6,100	20.00	30 0	Broken in 2 min.
C-4-a-750	750	3,600	12.50	76.0	Broken in 1 min.

hain and Ewen.<sup>6</sup> Beilby's amorphous theory in strain-hardened metals postulates the generation of amorphous metal at the planes of slip within the grains themselves during plastic deformation. That deformation takes place by slippage on cleavage and gliding planes within a grain<sup>7</sup> has

<sup>6</sup> W. Rosenhain and D. Ewen: Intercrystalline Cohesion in Metals. *Journal, Institute of Metals* (No. 2, 1912) 8, 149-173.

<sup>7</sup> J. A. Ewing and W. Rosenhain: The Crystalline Structure of Metals. *Phil. Trans. Royal Society of London* (1900) A, 193, 353.

F. Osmond and G. Cartaud: The Crystallography of Iron. *Journal, Iron and Steel Institute* (No. 3, 1906) 71, 447. Footnote continued on next page.

TABLE 5.—*Mechanical Properties at Various Temperatures of Copper Wire (C-5) Drawn at 150° C. from 0.075 to 0.025 In.*

Test Mark	Temp., ° C.	Tensile Strength, lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-5-a-L	-190	77,500	7.85	55.0	Tested in liquid air.
C-5-b-L	-190	74,600	17.25	60.0	
C-5-c-L	-190	73,500	14.10	60.0	
Average..	.....	75,500	13.00	58.0	
C-5-a-R	Room	57,200	3.90	76.0	
C-5-b-R	Room	56,100	3.05	69.0	
Average..	.....	56,650	3.50	72.5	
C-5-a-100	100	50,000	2.35	76.0	Tested in boiling water.
C-5-b-100	100	50,000	1.20	69.0	
Average..	.....	50,000	1.75	72.5	
C-5-a-200	215	40,000	1.55	82.0	Tested in hot crisco.
C-5-b-200	215	39,000	1.15	76.0	
Average..	.....	39,500	1.35	79.0	
C-5-a-330	320	40,000	1.55	80.0	Tested in electric furnace. Loaded rapidly.
C-5-b-330	320	40,000	1.55	80.0	
Average..	.....	40,000	1.55	80.0	
C-5-a-450	450	12,700	33.00	65.0	Broken in 30 sec.
C-5-b-450	450	9,200	20.25	30.0	Broken in 18 min.
C-5-a-600	600	5,600	18.75	21.0	Broken in 2 min.
C-5-a-750	750	3,600	15.00	51.5	Broken in 1 min.

been definitely proved. The amorphous phase of a metal is supposed to be harder at low temperatures than the crystalline phase, so the generation of more amorphous material by cold working would account for the increased hardness that is observed.

7 F. Robin: "Traité de Métallographie," 153. Paris, 1912. A. Hermann et fils.

Walter Rosenhain: Plastic Yielding of Iron and Steel. *Journal, Iron and Steel Institute* (No. 1, 1904) 65, 349.

H. M. Howe: "Metallography of Steel and Cast Iron." See particularly pp. 259-460. New York, 1916. McGraw-Hill Book Co., Inc.

TABLE 6.—*Mechanical Properties at Various Temperatures of Copper Wire (C-6) Drawn at 150° C. from 0.042 to 0.025 In.*

Test Mark	Temp., ° C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-6-a-L	-190	77,600	11.00	67.0	Tested in liquid air.
C-6-b-L	-190	77,600	9.35	67.0	
Average..	.....	77,600	10.20	67.0	
C-6-a-R	Room	53,000	3.05	69.0	
C-6-b-R	Room	51,100	1.55	72.5	
C-6-c-R	Room	52,200	3.05	69.0	Tested in boiling water.
Average..	.....	52,100	2.55	70.0	
C-6-a-100	100	48,000	1.55	76.0	
C-6-b-100	100	47,000	1.55	76.0	
C-6-c-100	100	47,000	2.35	72.5	
Average..	.....	47,330	1.80	75.0	Tested in hot crisco.
C-6-a-200	214	41,000	1.15	76.0	
C-6-b-200	215	39,000	1.55	82.0	
Average..	.....	40,000	1.35	79.0	
C-6-a-330	320	40,000	2.35	80.0	
C-6-a-450	450	15,800	32.00	88.0	Broken in 15 sec.
C-6-b-450	450	11,700	26.50	41.0	Broken in 5 min.
C-6-a-600	600	5,600	16.50	21.0	Broken in 2 min.
C-6-a-750	750	3,100	16.00	18.0	Slow loading.

In the amorphous theory<sup>8</sup> applied to annealed metals it is assumed that a film of amorphous material surrounds each crystalline grain. The amorphous phase has properties different from the crystalline and consequently the properties of a fine-grained annealed metal will be different from those of a coarse-grained one. For the purpose of this discussion,

<sup>8</sup> It is interesting to note that in 1902, T. W. Richards (*Proceedings, American Academy Arts and Sciences*, **38**, No. 15, 439) suggested that the intercrystalline material in ice probably had a different arrangement of the atoms from the crystalline phase.

TABLE 7.—*Mechanical Properties at Various Temperatures of Copper Wire (C-7) Drawn at 150° C. from 0.031 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elongation in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-7-c-L	-190	63,300	12.50	69.0	Tested in liquid air.
C-7-d-L	-190	64,300	19.50	65.0	
C-7-e-L	-190	64,300	18.75	67.0	
C-7-f-L	-190	63,000	14.75	60.0	
C-7-g-L	-190	67,000	17.50	65.0	
Average..	.....	64,400	16.60	65.0	
C-7-a-R	Room	46,800	4.30	72.5	Tested in boiling water.
C-7-b-R	Room	46,800	4.30	69.0	
Average	.....	46,800	4.30	70.7	
C-7-a-100	100	41,000	1.95	76.0	
C-7-b-100	100	42,000	2.35	65.0	
Average..	...	41,500	2.10	70.5	
C-7-a-200	215	34,700	0.75	76.0	Tested in hot crisco.
C-7-b-200	215	34,700	1.55	76.0	
Average	...	34,700	1.15	76.0	
C-7-a-330	320	33,500	3.90	72.5	
C-7-b-330	320	32,500	1.55	69.0	
Average	.....	33,000	2.70	71.0	
C-7-a-450	450	12,700	13.50	30.0	Broken in 3.5 min.
C-7-a-600	600	6,100	15.00	18.0	Broken in 2 min.
C-7-a-750	750	3,600	20.00	24.0	Loaded slowly.

it will be assumed that the amorphous hypothesis as applied to both worked and annealed metals is valid.

The properties of any substance are controlled by: The properties of each constituent present, the quantity of each constituent, the arrangement of the constituents, and the condition of the junctions between the physically different constituents. If some condition, like the temperature, is changed then the properties of the substance will vary, in addition to the foregoing, with the changes in properties of the constituents with change in temperature or other changed condition.

TABLE 8.—*Mechanical Properties at Various Temperatures of Copper Wire (C-8) Drawn Cold from 0.125 to 0.025 In. and then Heated for 10 Min. to 150° C.*

Test Mark	Temp. °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-8-a-L	-190	74,500	9.40	65.0	Tested in liquid air.
C-8-b-L	-190	73,500	7.80	65.0	
Average..	.....	74,000	8.60	65.0	
C-8-a-R	Room	56,200	1.55	69.0	
C-8-b-R	Room	55,200	1.55	69.0	
Average..	...	55,700	1.55	69.0	
C-8-a-100	100	55,000	1.55	69.0	Tested in boiling water.
C-8-b-100	100	56,000	1.20	65.0	
C-8-c-100	100	55,000	0.75	65.0	
C-8-d-100	100	51,000	0.75	72.0	
Average..	.....	54,250	1.06	68.0	
C-8-a-200	214	46,000	0.00	72.5	
C-8-b-200	211	45,000	1.55	72.5	Tested in hot crisco.
Average..	.....	45,500	0.75	72.5	
C-8-a-330	330	43,000	1.55	72.5	
C-8-b-330	330	40,000	1.55	72.5	Tested in electric furnace. Loaded rapidly.
Average..	.....	41,500	1.55	72.5	
C-8-a-450	450	18,300	29.50	88.0	Broken in 10 sec.
C-8-b-450	450	10,700	15.00	30.0	Broken in 10 min.
C-8-c-450	450	11,700	26.50	41.0	Broken in 3 min.
C-8-a-600	600	6,100	18.75	24.0	Broken in 2 min.
C-8-a-750	750	4,600	28.00	99.5	Broken in 5 sec.

The properties at various temperatures of a metallic substance composed of a single metallographic component are controlled by: (1) The properties of the amorphous phase of the metal, (2) the properties of the crystalline phase, (3) the quantity of each phase, (4) the arrangement of each phase, including junctions of one phase with another, (5) the changes in properties of each phase and the changes in quantity and arrangement of each phase with change in temperature.

TABLE 9.—*Mechanical Properties at Various Temperatures of Copper Wire (C-9) Drawn at 200° C. from 0.031 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
C-9-a-L	-190	60,000	14.00	69.0	Tested in liquid air.
C-9-b-L	-190	60,000	13.25	65.0	
C-9-c-L	-190	.....	15.50	65.0	
C-9-d-L	-190	61,000	16.25	60.0	
Average..	.....	60,300	14.75	65.0	
C-9-a-R	Room	45,000	4.65	69.0	
C-9-b-R	Room	45,000	4.65	72.5	
Average..	.....	45,000	4.65	71.0	
C-9-a-100	100	37,000	1.55	76.0	Tested in boiling water.
C-9-b-100	100	39,000	3.15	65.0	
C-9-c-100	100	39,000	2.35	69.0	
Average..	.....	38,300	2.35	70.0	
C-9-a-200	205	33,500	3.12	69.0	Tested in hot crisco.
C-9-b-200	205	34,500	1.95	72.5	
Average..	.....	34,000	2.55	71.0	
C-9-a-330	330	30,500	2.35	....	Tested in electric furnace. Loaded rapidly.
C-9-b-330	330	30,500	1.55	72.5	
Average..	.....	30,500	1.90	72.5	
C-9-a-450	450	11,200	17.75	30.0	Rapid loading.
C-9-b-450	450	10,200	14.90	18.0	Slow loading.
C-9-a-600	600	6,100	15.00	18.0	Broken in 2 min.
C-9-a-750	750	5,600	22.50	98.0	Broken in 3 sec.
C-9-b-950	950	710	3.10	5.0	Broken in 3 min.
C-9-b-950	950	2,500	7.80	11.0	Broken in 5 sec.

#### QUANTITY AND ARRANGEMENT OF AMORPHOUS PHASE

Little can be said regarding the actual quantities of the amorphous and crystalline phases in single component metals. The general arrangement and relative quantities of the two phases can be ascertained. In general, the quantity of the amorphous phase increases as the grain size



TABLE 10.—*Mechanical Properties at Various Temperatures of Tungsten Wire (T-1) Swaged and Drawn Hot from 0.165 to 0.025 In. Temperature Lowered from 1300° to 1000° During Working*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent	Reduction of Area, Per Cent	Remarks
T-1-a-L	-190	163,000	0 00	0 00	Tested in liquid air.
T-1-c-L	-190	177,000	0 00	0 00	
Average..	.....	170,000	0 00	0 00	
T-1-a-R	Room	234,000	3 15	10 0	
T-1-b-R	Room	233,000	3 15	18 0	
Average.	.....	233,500	3 15	14.0	
T-1-a-100	100	193,000	5 50	30 0	Tested in boiling water.
T-1-b-100	100	193,000	6.25	35 0	
Average	...	193,000	5 90	32 5	
T-1-a-200	207	161,000	4 30	45 0	Tested in hot crisco.
T-1-b-200	203	163,000	5 05	45 0	
Average..	....	162,000	4 65	45 0	
T-1-a-330	330	141,000	3 50	65 0	Tested in electric furnace.
T-1-b-330	330	145,000	3 50	65 0	
Average .	.....	143,000	3 50	65 0	
T-1-a-460	460	132,000	2.35	69 0	
T-1-a-600	600	128,000	1 55	69 0	
T-1-b-600	600	126,000	2 35	69 0	
Average..	.....	127,000	1 90	69 0	
T-1-a-750	750	112,000	1.55	72 5	
T-1-a-825	825	107,000	1 55	76 0	

decreases, as the amount of deformation increases, and as the temperature of deformation decreases.

The amorphous phase in annealed metals is between the grains. As the grain size decreases the grain-boundary surface increases and hence the quantity of the amorphous phase increases. When a metal is cold worked, amorphous metal is generated within the grains at the planes

TABLE 11.—*Mechanical Properties at Various Temperatures of Tungsten Wire 0.025 In. Diam. (T-2) Equiaxed*

Test Mark	Temp., ° C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
T-2-a-L	-190	55,000	0 00	0 00	Tested in liquid air.
T-2-b-L	-190	59,000	0 00	0 00	
T-2-c-L	-190	65,500	0 00	0 00	
Average..	.....	59,830	0.00	0 00	
T-2-a-R	Room	173,500	0 00	0 00	
T-2-b-R	Room	163,000	0 00	0 00	Tested in boiling water.
Average..	.....	168,250	0 00	0 00	
T-2-a-100	100	148,000	0.00	0 00	
T-2-b-100	100	158,000	0 00	0.00	
Average..	.....	153,000	0.00	0 00	
T-2-a-200	213	104,000	2 75	11.0	Tested in hot crisco.
T-2-b-200	213	102,000	7.75	11 0	
T-2-c-200	205	110,000	15.50	15.0	
T-2-d-200	214	98,000	9.40	11.0	
Average..	.....	103,500	8 85	12.0	
T-2-b-330	330	94,000	23 50	49.0	Tested in electric furnace.
T-2-c-330	330	98,000	20.25	60 0	
Average..	.....	96,000	21.80	54.5	
T-2-a-450	450	83,500	12.50	65.0	
T-2-b-450	450	82,500	15.50	65.0	
Average..	.....	83,000	14.00	65.0	
T-2-a-480	480	80,000	12.50	72.5	
T-2-b-600	600	67,500	20.00	69.0	
T-2-a-750	750	58,000	12.50	69.0	
T-2-b-750	750	57,000	14.00	69.0	
T-2-c-750	750	53,000	22.00	76.0	
Average..	.....	56,000	16.20	71.0	
T-2-a-835	835	55,000	20.30	60.0	
T-2-a-900	900	47,000	7.80	41.0	

TABLE 12.—*Mechanical Properties at Various Temperatures of Tungsten Wire (T-3) Drawn at 1000° C. from 0.125 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
T-3-a-L	-190	136,500	0.00	0.00	Tested in liquid air.
T-3-b-L	-190	157,000	0.00	0.00	
Average..	.....	146,750	0.00	0.00	
T-3-a-R	Room	232,000	6.25	35.0	
T-3-b-R	Room	232,000	6.25	35.0	Tested in boiling water.
T-3-c-R	Room	234,000	5.85	35.0	
Average..	.....	232,670	6.10	35.0	
T-3-a-100	100	193,000	7.00	41.0	
T-3-b-100	100	193,000	7.00	41.0	Tested in hot crisco.
Average..	.....	193,000	7.00	41.0	
T-3-a-200	207	159,000	7.40	45.0	
T-3-b-200	209	160,000	4.70	56.0	
T-3-c-200	216	160,000	6.25	51.0	Tested in electric furnace.
Average..	.....	159,670	6.10	50.7	
T-3-a-330	330	143,000	4.25	65.0	
T-3-b-330	330	143,000	4.25	65.0	
Average..	.....	143,000	4.25	65.0	
T-3-a-490	490	133,000	2.75	65.0	
T-3-a-600	600	123,000	2.75	69.0	
T-3-a-740	740	118,000	1.55	69.0	
T-3-a-850	850	102,000	1.55	69.0	
T-3-a-890	890	101,000	1.55	69.0	

of slip. In cold-worked metals, therefore, amorphous metal exists both at the grain boundaries and within the deformed grains.

The quantity of the amorphous phase will lessen when grain growth takes place; the quantity seems to be much greater within a severely deformed grain than at the grain boundary. A marked change in quantity and arrangement of amorphous metal, therefore, takes place when a cold-worked metal recrystallizes. This will cause the marked change in

TABLE 13.—*Mechanical Properties at Various Temperatures of Tungsten Wire (T-4) Drawn at 1000° C. from 0.042 to 0.028 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
T-4-a-L	-190	76,500	0 00	0.00	Tested in liquid air.
T-4-b-L	-190	101,000	0.00	0.00	
T-4-c-L	-190	73,500	0.00	0.00	
Average..	.....	83,670	0 00	0.00	
T-4-a-R	Room	190,000	0.00	0.00	
T-4-b-R	Room	194,000	0.00	0.00	Tested in boiling water.
Average..	.....	192,000	0.00	0.00	
T-4-a-100	100	161,000	1.55	2.0	
T-4-b-100	100	161,000	0.75	2.0	
Average..	.....	161,000	1.10	2.0	
T-4-a-200	216	128,000	12.50	53.0	Tested in hot crisco.
T-4-b-200	216	128,000	12.50	53.0	
Average..	.....	128,000	12.50	53.0	
T-4-a-330	330	111,000	6.25	64.5	
T-4-b-330	330	111,000	5.55	68.0	
T-4-c-330	330	108,000	4.70	61.0	Tested in electric furnace.
Average..	.....	110,000	5.50	64.4	
T-4-a-450	450	103,000	3.90	68.0	
T-4-a-650	650	94,000	3.10	68.0	
T-4-a-750	750	85,000	2.35	68.0	

properties observed between cold-worked and annealed metals at any given temperature.

#### PROPERTIES OF AMORPHOUS AND CRYSTALLINE PHASES

It has generally been assumed that the amorphous phase of a metal is weaker than the crystalline phase at higher temperatures but stronger at low temperatures. If this is true, there must be some temperature at which the strength or cohesion of the two phases is equal. The author has demonstrated this experimentally.<sup>9</sup> It has been found that the

<sup>9</sup>The Amorphous Metal Hypothesis and Equi-Cohesive Temperatures. *Journal, American Institute of Metals* (1917) 11, 300-324.

TABLE 14.—*Special Tests on Brittle Equiaxed Tungsten (T-2) Wire*

Test Mark	Breaking Load, Lb. per Sq. In.	Remarks
T-2-a-S	120,000	Stressed to 101,000 lb. per sq. in. at room temperature and then immersed under stress in liquid air and broken.
T-2-b-S	177,000	Stressed to 137,000 lb. per sq. in. at room temperature and then immersed under stress in liquid air and broken.
T-2-c-S	73,500	Stressed to 8000 lb. per sq. in. at room temperature and immersed while under stress in liquid air and broken.
T-2-d-S	177,000	Stressed to 151,000 lb. per sq. in. at room temperature and immersed while under stress in liquid air and broken.
T-2-e-S	60,000	Not stressed at room temperature. Tested in liquid air in usual way.

amorphous and crystalline phases have the same cohesion at a temperature corresponding very nearly to the lowest recrystallization temperature of the particular metal after severe cold working. I have called this the equi-cohesive temperature. Above the equi-cohesive temperature, the amorphous phase has less cohesion, and below, greater cohesion than the crystalline phase. The cohesion in each case is measured by the load that will cause permanent deformation; any load that will permanently deform the amorphous phase will eventually break it if time is given, because no new stronger phase is formed by the deformation and a load that permanently deforms the crystalline phase changes its cohesion.

Wires of tungsten, iron, gold, silver, and platinum were prepared in both the coarse- and fine-grained states. These were tested by shaping into the form of hair pins and clamping the two ends in such a manner that they served as the fulcrum of the cantilever. Weights were hung on the loop ends of the hair pins and the history of deformation with change in temperature was obtained.

It was found that the metals in the fine-grained state offered the greater resistance to deformation at low temperatures and that at high temperatures they offered less resistance to deformation than the metals in the coarse-grained state. At an intermediate temperature for each metal, the resistance to deformation (cohesion) was independent of grain size. Since a variation in the quantity of the amorphous phase

TABLE 15.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-1) Drawn Cold from 0.120 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-1-b-L	-190	166,500	1 95	51 0	Tested in liquid air.
I-1-c-L	-190	164,300	1 55	41.0	
Average.	.....	165,400	1 75	46 0	
I-1-a-R	Room	97,500	1.50	60 0	
I-1-b-R	Room	96,000	0.80	72.0	
I-1-c-R	Room	95,000	1 50	69.0	Tested in boiling water.
Average..	.....	96,170	1.30	67 0	
I-1-a-100	100	95,000	0.00	69 0	
I-1-b-100	100	. . .	0 35	72.5	
I-1-c-100	100	86,500			
I-1-d-100	100	97,000			Tested in hot crisco.
Average.	.....	92,800	0.20	70 7	
I-1-a-200	207	90,000	1.55	65 0	
I-1-b-200	205	90,000	0.75	65 0	
Average.	.....	90,000	1 15	65 0	
I-1-a-330	330	80,000	1 55	51 5	Tested in electric furnace.
I-1-b-330	330	80,000	2 35	60 0	
Average .	.....	80,000	1 95	55 5	
I-1-a-450	450	51,000	5.50	72 5	
I-1-b-450	455	50,000	3 90	72 5	
Average.	.. . .	50,500	4.70	72 5	Annealed during test.
I-1-a-575	575	18,000	22.00	82.5	
I-1-b-575	550	26,000 to 16,000	11.00	88.0	
I-1-c-500	500	41,000	7.00	76.0	
I-1-a-690	690	6,400	Not taken	51.0	
I-1-a-735	735	5,100		51.0	Alpha iron.
I-1-a-850	850	2,550		61.0	Beta iron.
I-1-b-850	850	2,040		69.0	
I-1-a-940	940	2,550		41.0	Gamma iron.
I-1-b-940	940	2,860		41.0	
I-1-c-960	960	4,100	5.00		

TABLE 16.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-2) 0.025 In. Diam. Annealed at 800° C.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elongation in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-a-b-L	-190	108,100	1.55	50.0	Tested in liquid air.
I-a-c-L	-190	114,000	0.75	0.0	
I-2-d-L	-190	112,000	0.75	4.0	
I-2-e-L	-190	104,000	0.75	14.0	
Average..	.....	109,500	0.96	17.0	
I-2-a-R	Room	49,000	12.50	79.0	
I-2-b-R	Room	48,100	12.50	72.5	
Average..	.....	48,550	12.50	75.7	
I-2-a-100	100	52,000	7.00	72.5	
I-2-b-100	100	52,000	6.25	76.0	
I-2-c-100	100	50,000	4.70	65.0	Tested in boiling water.
I-2-d-100	100	51,000	4.70		
Average..	.....	51,250	5.66	71.0	
I-2-a-200	206	61,000	10.10	69.0	
I-2-b-200	207	60,000	11.50	51.0	
I-2-c-200	211	57,000	10.50	56.0	Tested in hot crisco.
Average..	.....	59,330	10.50	59.0	
I-2-a-330	330	52,000	11.00	55.0	
I-2-b-330	330	52,000	11.00	51.0	
Average..	.....	52,000	11.00	53.0	
I-2-a-450	450	26,500	24.00	69.0	Tested in electric furnace.
I-2-b-450	460	27,500	36.00	55.0	
Average..	.....	27,000	30.00	62.0	
I-2-a-575	575	14,000	17.00	41.0	
I-2-b-575	575	14,000	22.00	60.0	
Average..	.....	14,000	19.50	50.5	

did not vary the cohesion, I assume that the cohesion of the amorphous and crystalline phases must be the same at what I have called the equi-cohesive temperature.

It is not to be understood that the mechanical properties of a metal are independent of grain size at the equi-cohesive temperature, because

TABLE 17.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-3) Drawn Cold from 0.042 to 0.025 In.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-3-b-L	-190	146,800	1.55	60.0	Tested in liquid air.
I-3-c-L	-190	145,800	1.55	51.0	
Average..	.....	146,300	1.55	56.0	
I-3-a-R	Room	89,000	1.50	72.5	
I-3-b-R	Room	86,800	1.10	69.0	
I-3-c-R	Room	84,800	4.65	69.0	Tested in boiling water.
I-3-d-R	Room	84,000	3.10	69.0	
I-3-e-R	Room	84,000	2.75	72.5	
Average.	.....	85,720	2.60	70.0	
I-3-a-100	100	72,500	2.35	69.0	
I-3-b-100	100	74,500	2.35	69.0	Tested in hot crisco.
I-3-c-100	100	81,500	2.35	69.0	
I-3-d-100	100	83,500	1.55	72.5	
Average..	.....	78,000	2.15	70.0	
I-3-a-200	213	82,500	1.55	72.5	
I-3-b-200	214	80,500	1.55	69.0	Tested in electric furnace.
Average..	.....	81,500	1.55	71.0	
I-3-a-330	330	75,000	5.50	69.0	
I-3-b-330	330	75,000	9.00	65.0	
I-3-c-330	330	77,500	7.80	69.0	
Average..	.....	75,800	7.40	68.0	Annealed during test.
I-3-a-450	450	47,000	7.00	76.0	
I-3-b-450	450	47,000	5.50	76.0	
Average..	.....	47,000	6.25	76.0	
I-3-a-500	500	41,000	7.80	76.0	
I-3-a-575	575	21,000	11.70	80.0	

nothing could be farther from the truth. The tensile strength, elongation, and reduction of area vary in a remarkable manner at and near the equi-cohesive temperature with variation in grain size. This variation is caused partly by the variations in quantity and arrangement of the amorphous phase due to differences in grain size and partly by the fact



TABLE 18.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-4) Drawn Cold from 0.031 to 0.025 In. Diam.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-4-a-L	-190	146,800	1.15	4.0	Tested in liquid air.
I-4-b-L	-190	142,700	1.55	0 0	
Average..	.....	144,750	1.35	2 0	
I-4-a-R	Room	69,500	4.30	51.0	
I-4-b-R	Room	70,400	2.75	55.0	
Average..	.....	69,950	3.50	53.0	
I-4-a-100	100	68,500	2.35	65.0	Tested in boiling water.
I-4-b-100	100	73,500	2.35	60.0	
Average..	.....	71,000	2.35	62.5	
I-4-a-200	215	72,500	1.55	69.0	Tested in hot crisco.
I-4-b-200	213	72,500	1.15		
Average..	.....	72,500	1.35	69.0	
I-4-a-330	330	73,500	4.70	60.0	Tested in electric furnace.
I-4-b-330	330	67,500	5.50	51.5	
Average..	.....	70,500	5.10	55.7	
I-4-a-450	445	39,000	7.80	72.5	
I-4-b-450	450	39,000	7.00	72.5	
Average..	.....	39,000	7.40	72.5	
I-4-a-500	500	33,000	4.70	41.0	
I-4-a-545	545	27,000	7.80	41.0	
I-4-a-575	575	21,000	7.80	41.0	
					Annealed during test.

that the amorphous phase deforms at a very much slower rate under given conditions than the crystalline grains.<sup>10</sup> Also the directional properties of the grains are less manifest as the grain size decreases.

The difference in rate of deformation of the amorphous metal and

<sup>10</sup> The deformation of the crystalline grains is assumed to take place by movement at slip planes and not by actual permanent change of each atom with reference to its neighbors.

TABLE 19.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-5) Drawn at 400° C. from 0.075 to 0.025 In. Diam.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-5-a-L	-190	157,100	0 75	65 0	Tested in liquid air.
I-5-b-L	-190	153,000	1 55	51 0	
Average	.....	155,050	1 15	58 0	
I-5-a-R	Room	96,000	1 95	60 0	
I-5-b-R	Room	98,000	....	60 0	
I-5-c-R	Room	100,000	2 35	60 0	Tested in boiling water.
Average	..	98,000	2 15	60 0	
I-5-a-100	100	93,000	2 35	60 0	
I-5-b-100	100	99,000	1 15	60 0	
I-5-c-100	100	97,000	0 75	60 0	
Average..	.....	96,330	1 40	60 0	Tested in hot crisco.
I-5-a-200	214	101,000	1 15	51.5	
I-5-b-200	213	99,000	1.15	60 0	
Average.		100,000	1.15	56 0	
I-5-a-330	330	88,000	4.70	51 5	
I-5-b-330	330	90,000	4 70	60 0	Tested in electric furnace.
Average		89,000	4 70	56 0	
I-5-a-450	450	59,000	3 10	69 0	
I-5-b-450	450	58,000	3 90	69 0	
Average.	.	58,500	3 50	69.0	
I-5-a-500	500	48,000	3 90	76 0	Annealed during test. Slow loading.
I-5-b-500	500	50,000	4.70	76 0	
Average	...	49,000	4 30	76 0	
I-5-a-575	575	28,000 to 18,000	18 00	80 0	
I-5-b-575	575	20,000	7 80	....	
I-5-c-575	575	27,000	11.70	76 0	Rapid loading.
I-5-a-650	650	11,200	38 00	69 0	Broken in 10 sec.
I-5-b-650	650	7,100	29 00	41.0	Broken in 7 min.
I-5-a-725	725	7,100	32.00	56.0	Broken in 10 sec.
I-5-b-725	725	4,600	18 50	36 0	Broken in 5 min.
I-5-a-835	835	4,100	43.50	65.0	Broken in 10 sec.
I-5-b-835	835	2,550	23 50	41.0	Slow loading.
I-5-a-950	950	5,100	9.00	30 0	Fast loading.
I-5-a-1000	1000	3,100	7.50	35.0	Slow loading.

TABLE 20.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-6) Drawn at 400° C. from 0.042 to 0.025 In. Diam.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-6-a-L	-190	135,500	0.35	10.0	Tested in liquid air.
I-6-b-L	-190	143,000	0.75		
Average..	.....	139,250	0.55	10.0	
I-6-a-R	Room	88,800	1.55	30.0	
I-6-b-R	Room	100,000	1.55	51.0	Tested in boiling water.
I-6-c-R	Room	92,000	0.75	23.0	
Average..	.....	93,600	1.30	33.0	
I-6-a-100	100	90,000	0.75	41.0	
I-6-b-100	100	86,500	1.15	41.0	Tested in hot crisco.
Average..	.....	88,250	0.90	41.0	
I-6-a-200	215	86,500	0.75	51.0	
I-6-b-200	214	97,000	1.55	60.0	
I-6-c-200	211	91,000	0.75	41.0	Tested in electric furnace.
Average..	.....	91,500	1.00	51.0	
I-6-a-330	330	81,500	4.70	41.0	
I-6-b-330	330	77,500	9.00	60.0	
I-6-c-330	330	79,500	3.10	45.0	Annealed during test.
Average..	.....	79,500	5.60	49.0	
I-6-a-450	450	47,000	3.10	55.0	
I-6-b-450	450	43,000	6.25	76.0	
I-6-c-450	450	48,000	7.00	76.0	Annealed during test.
Average..	.....	46,000	5.45	69.0	
I-6-a-575	575	14,000	23.50	69.0	
I-6-b-575	575	16,000	10.00	60.0	
Average..	.....	15,000	16.75	64.5	

crystalline grains is shown strikingly by hardness tests on lead and tin at room temperature, which is above the equi-cohesive temperatures of both of these metals. A piece of cast lead was cut in two and half of it was flattened by cold hammering. Recrystallization of the cold-hammered lead took place at room temperature; the large grains that com-

TABLE 21.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-7) Drawn at 400° C. from 0.031 to 0.025 In. Diam.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-7-a-L	-190	135,000	0 35	15.0	Tested in liquid air.
I-7-b-L	-190	133,500	0.00	45.0	
Average..	.....	134,250	0.20	30.0	
I-7-a-R	Room	74,500	3.85	60 0	
I-7-b-R	Room	74,500	6.25	65.0	Tested in boiling water.
I-7-c-R	Room	70,400	4.70	51 0	
Average..	.....	73,100	4.90	59.0	
I-7-a-100	100	78,500	0 75	60.0	
I-7-b-100	100	83,500	0.35	60.0	Tested in hot crisco.
Average..	.....	81,000	0 55	60.0	
I-7-a-200	208	74,000	0 75	51 5	
I-7-b-200	203	75,000	0.75	60 0	
Average..	.....	74,500	0.75	56 0	Tested in electric furnace.
I-7-b-330	330	74,500	2.35	45.0	
I-7-c-330	330	74,500	3.10	51 0	
Average..	.....	74,500	2.75	48 0	
I-7-a-450	450	45,000	7.80	76 0	Tested in electric furnace.
I-7-b-450	450	45,000	7.80	76.0	
Average..	.....	45,000	7.80	76.0	
I-7-a-575	575	22,000	6.25	51.0	
I-7-b-575	575	22,000	7.50	41.0	Tested in electric furnace.
Average..	.....	22,000	6.90	46.0	
I-7-a-725	725	7,100	22.50	55.0	
I-7-b-725	725	4,600	15.50	24.0	
I-7-a-835	835	4,100	19.00	51.0	Started loading slow and finished fast.
I-7-b-835	835	2,550	20.00	30.0	
I-7-a-950	950	3,250	6.25	6.0	Loaded slowly.
I-7-b-950	950	5,100	10.00	18.0	

TABLE 22.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-8) Drawn Cold from 0.120 In. to 0.025 In. Diam. and then Heated 10 Min. to 400° C.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elongation in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-8-a-L	-190	137,000	2.75	64.0	Tested in liquid air.
I-8-b-L	-190	137,000	3.10	64.0	
Average..	.....	137,000	2.90	64.0	
I-8-a-R	Room	70,400	3.95	75.0	
I-8-b-R	Room	70,400	4.70	69.0	Tested in boiling water.
Average..	....	70,400	4.30	72.0	
I-8-a-100	100	69,500	3.90	72.0	
I-8-b-100	100	69,500	1.55	76.0	
Average..	.....	69,500	2.70	74.0	Tested in hot crisco.
I-8-a-200	210	74,000	1.95	69.0	
I-8-b-200	210	74,000	2.35	69.0	
Average..	.....	74,000	2.15	69.0	
I-8-a-330	330	66,500	20.30	72.5	Tested in electric furnace.
I-8-b-330	330	66,500	17.10	65.0	
I-8-c-330	320	69,500	12.50		
Average..	.....	67,500	16.60	69.0	
I-8-a-450	450	44,000	4.70	82.0	
I-8-b-450	450	43,000	6.25	76.0	
I-8-c-450	450	45,000	5.50		
Average..	.....	44,000	5.50	79.0	
I-8-a-575	575	15,000	28.00	82.5	Annealed during test.
I-8-b-575	575	15,000	30.00	82.5	Annealed during test.
Average..	.....	15,000	29.00	82.5	

posed the cast metal were broken up into small equiaxed grains. In determining the Brinell hardness of the coarse-grained and fine-grained lead, it was found that both would deform under constant load for a considerable time. The hardness of the cast lead immediately after the beam of the Brinell machine first registered the full load was 5.44, while that of the cold-hammered lead under the same conditions was

TABLE 23.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-9) Drawn at 275° C. From 0.042 In. to 0.025 In. Diam.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elongation in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-9-a-L	-190	168,000	....	30 0	Tested in liquid air.
I-9-b-L	-190	167,000	....	30 0	
Average..	....	167,500	....	30 0	
I-9-a-R	Room	106,000	1 55	69 0	
I-9-b-R	Room	116,000	1 55	65 0	
Average..	.....	111,000	1 55	67 0	
I-9-a-100	100	107,000	2 35	69 0	Tested in boiling water.
I-9-b-100	100	107,000	1 95	72 5	
Average..	.....	107,000	2 10	71 0	
I-9-a-200	215	114,000	1 55	69 0	Tested in hot crisco
I-9-b-200	208	115,000	1 55	60 0	
Average .	.....	114,500	1 55	64 5	
I-9-a-330	330	106,000	4 70	51 0	Tested in electric furnace.
I-9-b-330	330	99,000	3 90	51 0	
I-9-c-330	330	101,000	4 70	60 0	
Average .	... .	102,000	4 40	54 0	
I-9-a-450	450	60,000	4 70	51 0	
I-9-b-450	450	60,000	4 70	69 0	
Average..	.....	60,000	4 70	60 0	
I-9-a-575	575	14,000	36 50	76 0	Annealed during test.
I-9-b-575	575	13,700	38 00	76 0	Annealed during test.

6.54. When the load was maintained constant until substantially no further deformation took place, the hardness of the cast lead was 5.10 and that of the cold-hammered lead 4.66. The cold-hammered lead had deformed more slowly but eventually its equilibrium amount of deformation was greater than that of the coarse-grained or cast lead. The same was true of the tin and the differences were even more marked. The hardness of cast tin when the load was first registered was 9.12 and that of the cold-hammered tin (which had also recrystallized at room temperature) was 11.32. When the hardness tests were continued

TABLE 24.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-10) Drawn at 275° C. From 0.031 In. to 0.025 In. Diam.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-10-a-L	-190	166,000	0.35	0.0	Tested in liquid air.
I-10-b-L	-190	164,000	....	4.0	
Average..	.....	165,000	0.35	2.0	
I-10-a-R	Room	96,000	1.55	55.0	
I-10-b-R	Room	100,000	1.55	65.0	
Average..	.....	98,000	1.55	60.0	
I-10-a-100	100	104,000	0.75	51.5	Tested in boiling water.
I-10-b-100	100	105,000	0.75	60.0	
Average..	.....	104,500	0.75	56.0	
I-10-a-200	210	101,000	0.75	60.0	Tested in hot crisco.
I-10-b-200	206	99,000	0.75	51.5	
Average..	.....	100,000	0.75	56.0	
I-10-a-330	330	95,000	3.12	51.5	Tested in electric furnace.
I-10-b-330	330	95,000	2.35	60.0	
Average..	.....	95,000	2.75	56.0	
I-10-a-450	450	51,000	5.50	55.0	
I-10-b-450	450	51,000	5.50	69.0	
Average..	.....	51,000	5.50	62.0	
I-10-a-575	575	17,000	17.00	69.0	Partly annealed during test.
I-10-b-575	575	15,000	27.00	69.0	
Average..	.....	16,000	22.00	69.0	

under constant load to no further deformation, the cast, or coarse-grained, tin had a hardness of 7.3 and the hammered, or fine-grained, tin, 4.72.

This viscosity is typical of amorphous materials. The amorphous phase deforms slowly because the deformation has to be transmitted from atom to atom. Any change in shape, in general, will involve the change of position of every atom relative to every other atom in the deformed region. This is not true in the deformation of crystalline material. Blocks of the crystalline material consisting of millions of atoms

TABLE 25.—*Mechanical Properties at Various Temperatures of Armco Iron Wire (I-12) 0.025 In. Diam. Annealed at 750° C.*

Test Mark	Temp., °C.	Tensile Strength, Lb. per Sq. In.	Elonga- tion in 2 In., Per Cent.	Reduction of Area, Per Cent.	Remarks
I-12-a-L	-190	104,000	13.30	56.0	Tested in liquid air.
I-12-b-L	-190	106,000	6.20	61.0	
I-12-c-L	-190	106,000	17.00	56.0	
I-12-d-L	-190	106,000	10.50	69.0	
Average..	.....	105,500	11.75	62.0	
I-12-a-R	Room	47,000	18.00	78.0	
I-12-b-R	Room	48,000	23.50	78.0	
I-12-c-R	Room	49,000	26.50	78.0	
I-12-d-R	Room	48,000	25.00	78.0	
I-12-e-R	Room	48,500	24.50	80.0	
Average..	.....	48,200	23.70	78.5	Tested in boiling water.
I-12-a-100	100	50,500	10.20	82.0	
I-12-b-100	100	48,500	10.20	81.0	
I-12-c-100	100	49,500	7.50	80.0	
Average..	.....	49,500	8.40	81.0	
I-12-a-200	220	60,500	13.25	79.0	Tested in hot crisco.
I-12-b-200	220	62,500	14.00	79.0	
Average..	.....	61,300	13.60	79.0	
I-12-a-330	330	57,500	17.20	63.0	Tested in electric furnace.
I-12-b-330	330	55,500	17.20	63.0	
Average..	.....	56,500	17.20	63.0	
I-12-a-450	450	30,000	35.00	72.0	Short heating. Clamps not up to temperature of furnace.
I-12-b-450	455	29,000	36.75	72.0	
Average..	.....	29,500	35.90	72.0	
I-12-a-575	575	13,000	19.50	53.0	
I-12-b-575	575	14,000	32.00	53.0	
I-12-a-735	735	4,200	29.00	40.0	
I-12-a-840	840	2,400			



move by displacement along cleavage or gliding planes and the atoms within one such block or fragment remain at the end of deformation in about the same positions, relative to one another, as they had before deformation. The atoms within one fragment, however, have changed

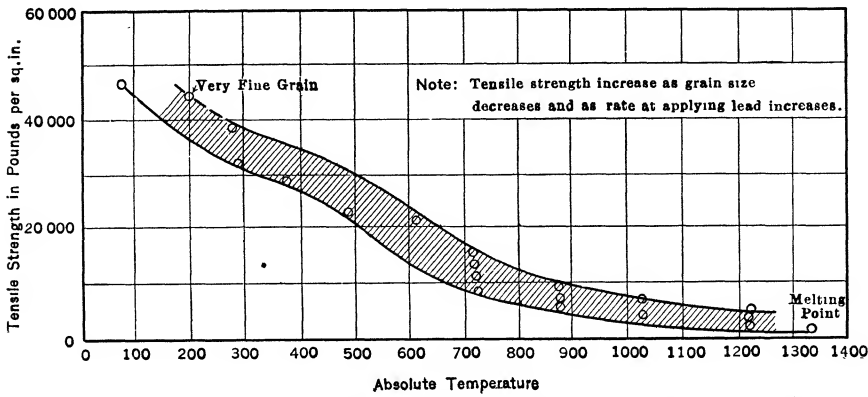


FIG. 43.—TENSILE STRENGTH OF ANNEALED COPPER WIRE 0.025 IN. DIAM. WITH VARIATIONS IN TEMPERATURE, GRAIN SIZE AND RATE OF LOADING.

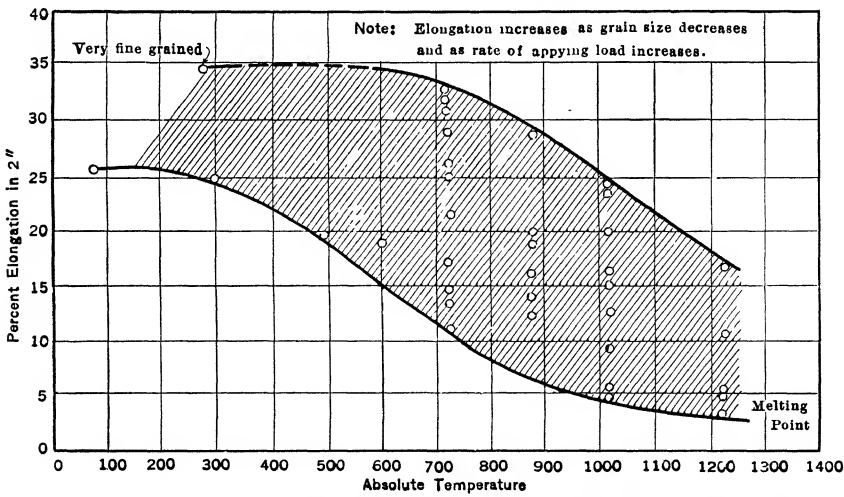


FIG. 44.—PER CENT. ELONGATION IN 2 IN. OF ANNEALED COPPER WIRE OF 0.025 IN. DIAM. WITH VARIATIONS IN TEMPERATURE, GRAIN SIZE AND RATE OF LOADING.

their positions with reference to atoms of another fragment. The movement of these relatively large blocks of atoms would take place much more rapidly than a movement in which each atom had to move relative to each other atom.

The quantity of deformation possible in an amorphous substance at relatively high temperatures should have only atomic dimensions as its

limit. The amount of deformation by tension has a definite limit but varies with the temperature and rate of loading. We are all familiar with the permanent deformation of glass tubes and rods that have been stored for months in a bent position; the bends become permanent in time. If an attempt is made to permanently deform the glass the same amount in a few seconds or even in a few hours, it will break. At high temperatures glass deforms more with rapid than with slow loading. When glass is in its soft-temperature range, "hairs" so small as to be scarcely visible to the naked eye can be produced by partly disengaging

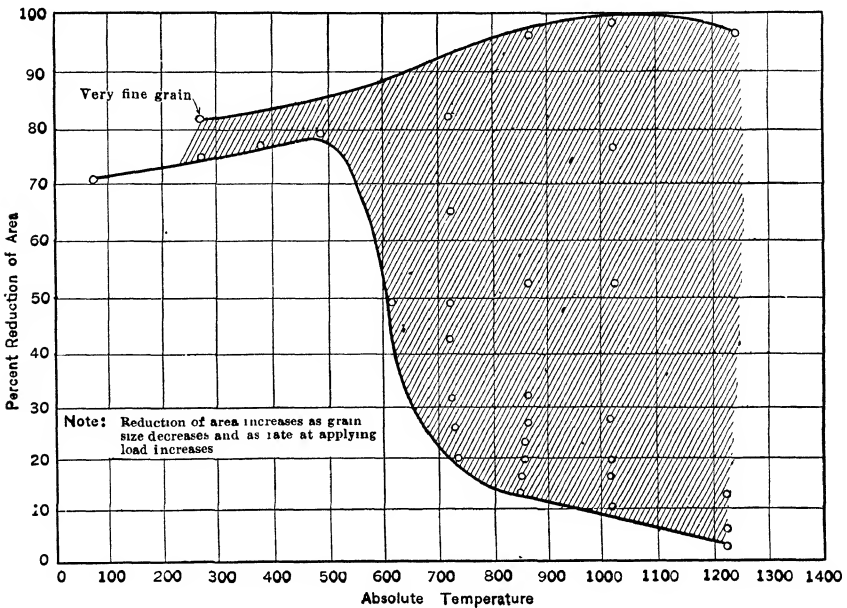


FIG. 45.—REDUCTION OF AREA OF ANNEALED COPPER WIRE 0.025 IN. DIAM. WITH VARIATIONS IN TEMPERATURE, GRAIN SIZE AND RATE OF LOADING.

one part from the main mass and removing it very rapidly. This extreme deformation of red-hot glass is largely due to the increase in strength of the small "hair" because of its lowered temperature. The main mass of the glass remains hot, and hence weak, and is drawn into a fine "hair" by the cooler glass thread. But glass broken at red heat at constant temperature deforms more by rapid than by slow loading. The same is true of sealing wax and pitch.

A few simple experiments with an amorphous material like celluloid illustrate the sluggish motion of the atoms with respect to permanent change in position. A celluloid strip was bent through an angle of  $180^\circ$  and released quickly; it returned to about its original position. It was bent  $180^\circ$  again and held there 1 hr. When it was released it sprang

quickly to within  $20^\circ$  of the original position and then changed slowly during several hours, after which the permanent deformation was about  $10^\circ$ . The greater part of the latter change occurs during the first 10

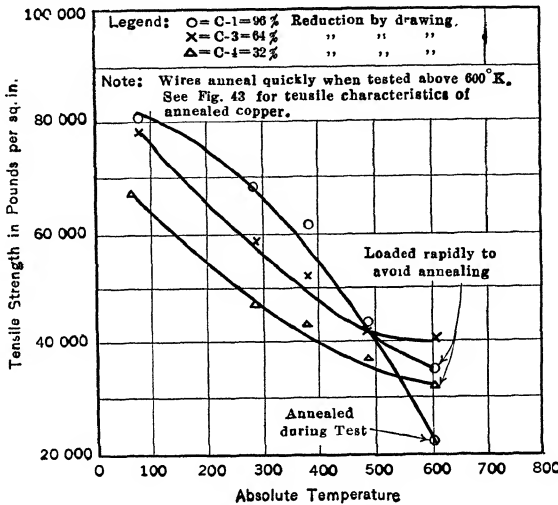


FIG. 46.—TENSILE STRENGTH OF COLD-DRAWN COPPER WIRES 0.025 IN. DIAM. AT TEMPERATURE BELOW THAT OF ANNEALING.

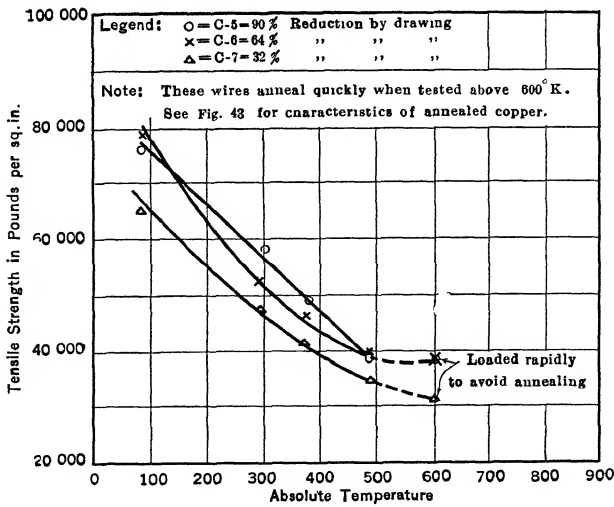


FIG. 47.—TENSILE STRENGTH BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM. DRAWN AT  $150^\circ\text{C}$ .

min. after releasing. Now if the strip is bent  $180^\circ$ , held 1 hr. and released and immediately bent  $180^\circ$  in the opposite direction and released, it springs, within 5 sec., to a position about  $15^\circ$  on the side of first bending

and then gradually moves in the direction of the second rapid bending until it assumes the equilibrium position ( $10^\circ$ ) after 1 hr. or more.

Similarly, when rubber is stretched quickly and released, there is little permanent stretch; but when it is stretched the same amount and

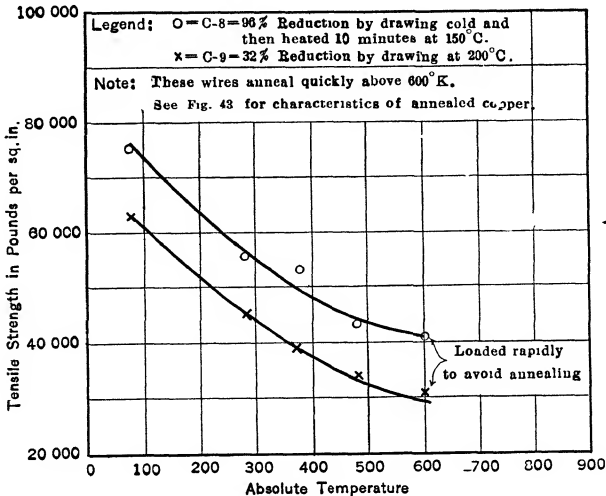


FIG. 48.—TENSILE STRENGTH BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM.

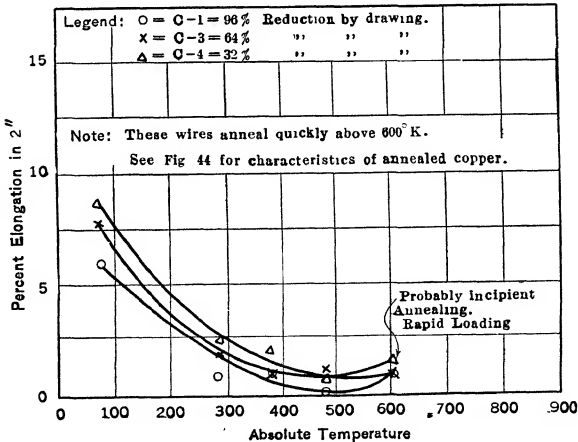


FIG. 49.—PER CENT. ELONGATION BELOW ANNEALING TEMPERATURE OF COLD-DRAWN COPPER WIRES 0.025 IN. DIAM.

held in tension for an extended period, considerable elongation, say 10 per cent., is noted when the tension is removed. Immediately after releasing the tension, however, the rubber begins to contract toward its original length and at the end of 30 min. the permanent stretch may

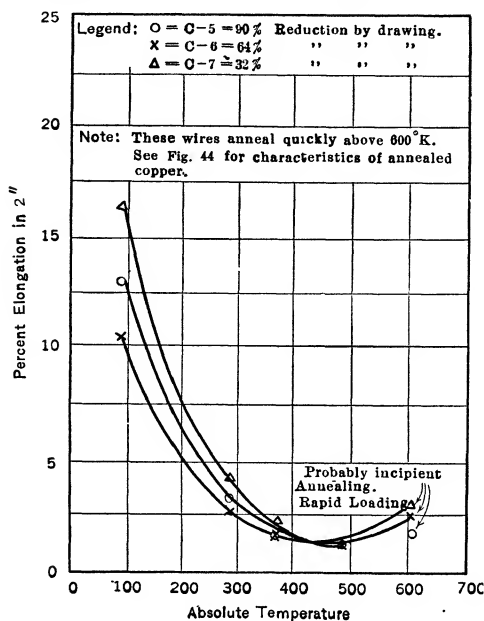


FIG. 50.—PER CENT. ELONGATION BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM. DRAWN AT 150° C.

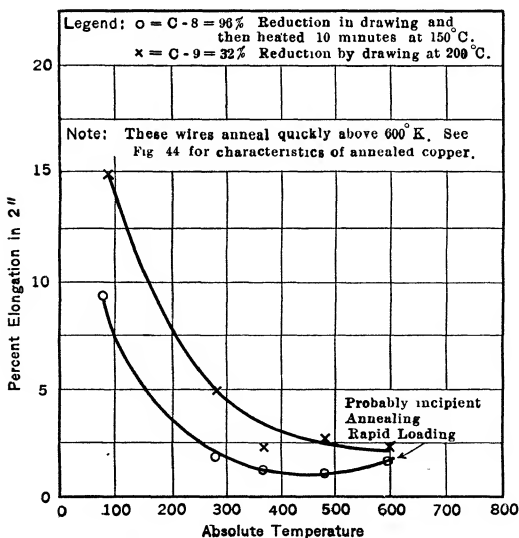


FIG. 51.—PER CENT. ELONGATION BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM.

have been reduced to, say, 5 per cent. Also, the rubber can hold for a short period a load that will break it in a longer time. Its cohesion, therefore, depends on the time of application of the load that measures it. This is

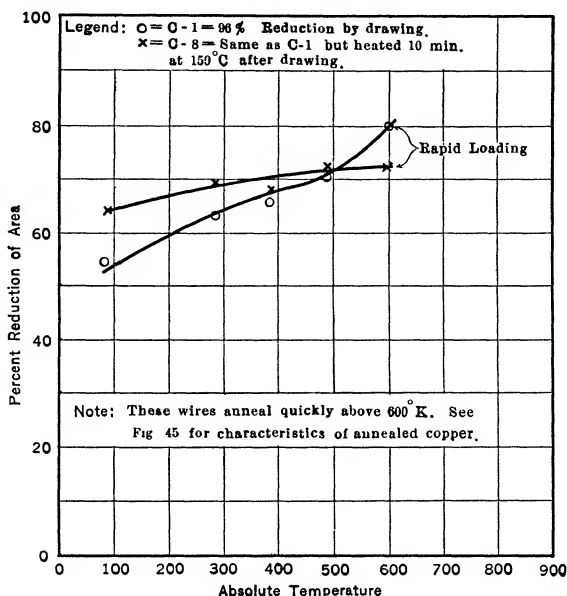


FIG. 52.—PER CENT. REDUCTION OF AREA OF COPPER WIRES 0.025 IN. DIAM. BELOW ANNEALING TEMPERATURE.

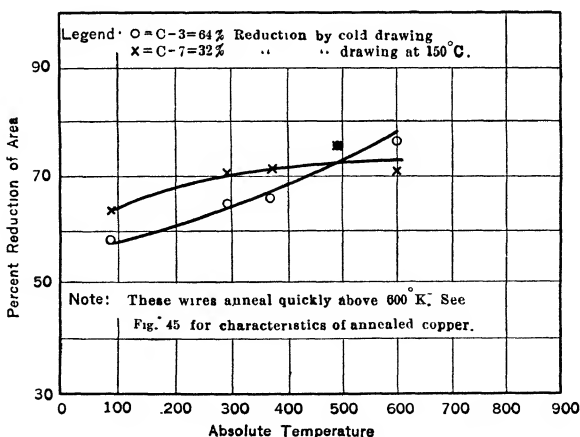


FIG. 53.—PER CENT. REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM.

typical of amorphous substances. All of the foregoing peculiarities should be kept in mind for the interpretation of the effect of the amorphous phase on the physical properties of metals.

It is not so much the quantity of deformation in the amorphous phase itself in metals that controls the total deformation but rather the distribution of stress that its slight deformation permits, thereby forcing the crystalline grains to deform.

One of the most important factors in the interpretation of the properties of metals with change of temperature is the differential change in

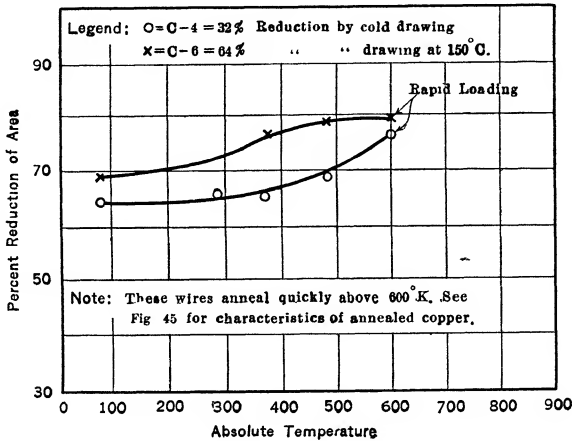


FIG. 54.—REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM.

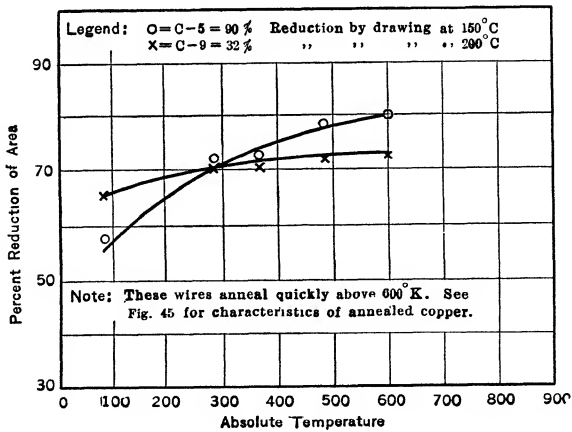


FIG. 55.—PER CENT. REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF COPPER WIRES 0.025 IN. DIAM.

cohesion between the amorphous and crystalline phases with change in temperature. In Fig. 74 is shown the general cohesion-temperature curves of the amorphous and crystalline phases of any material that may exist in both of these states. This particular curve shows no allotrope in the crystalline phase. So far as we know, no allotrope can occur in

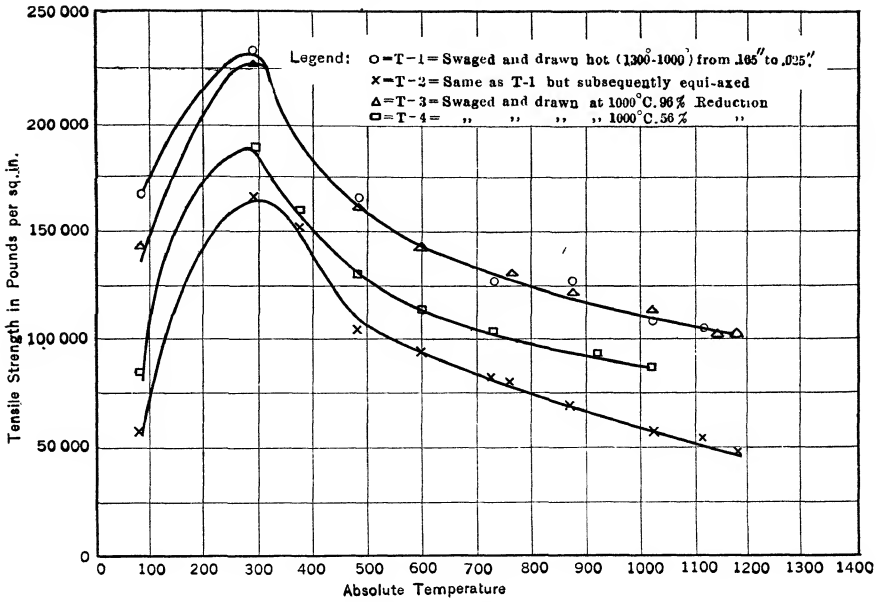


FIG. 56.—TENSILE STRENGTH AT VARIOUS TEMPERATURES OF TUNGSTEN WIRES.

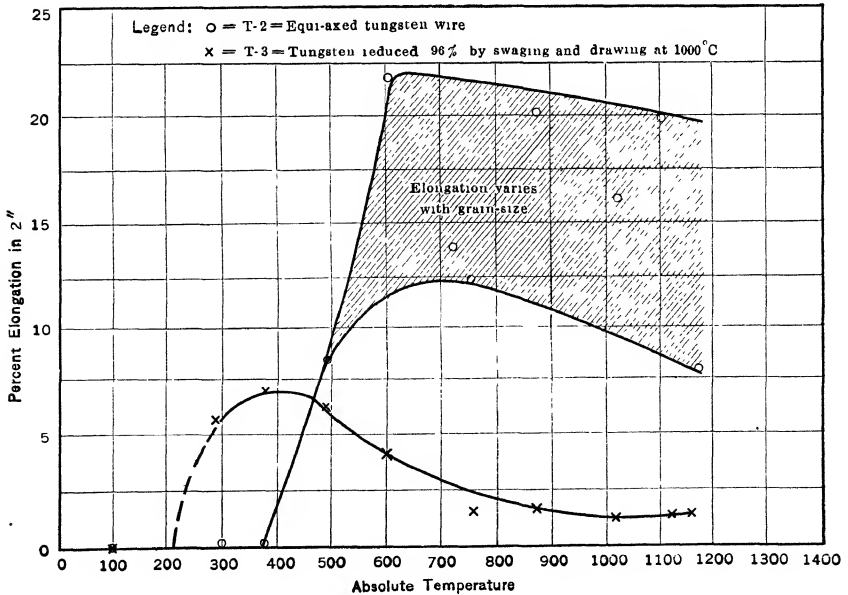


FIG. 57.—PER CENT. ELONGATION OF TUNGSTEN WIRES 0.025 IN. DIAM. AT VARIOUS TEMPERATURES.



the amorphous phase, since allotropy is one of the results of crystallinity; if substances were not crystalline they would not be allotropic. The occurrence of allotropy, therefore, in a crystalline material would alter the cohesion-temperature curve in the crystalline phase only. If allotropy proves to be caused by a change within the atom, this view will

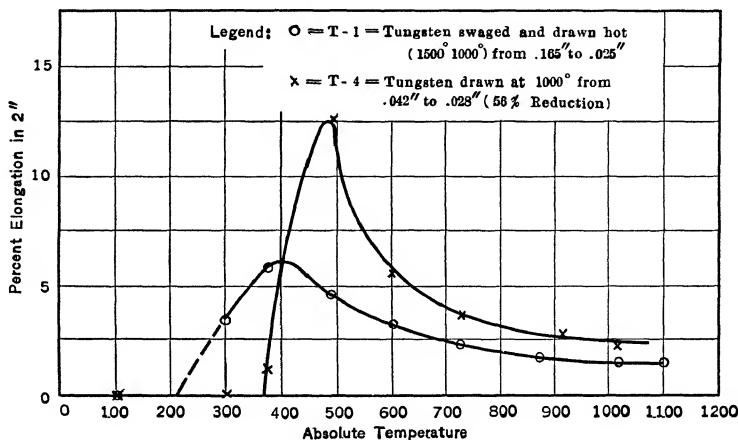


FIG. 58.—PER CENT. ELONGATION OF TUNGSTEN WIRES AT VARIOUS TEMPERATURES.

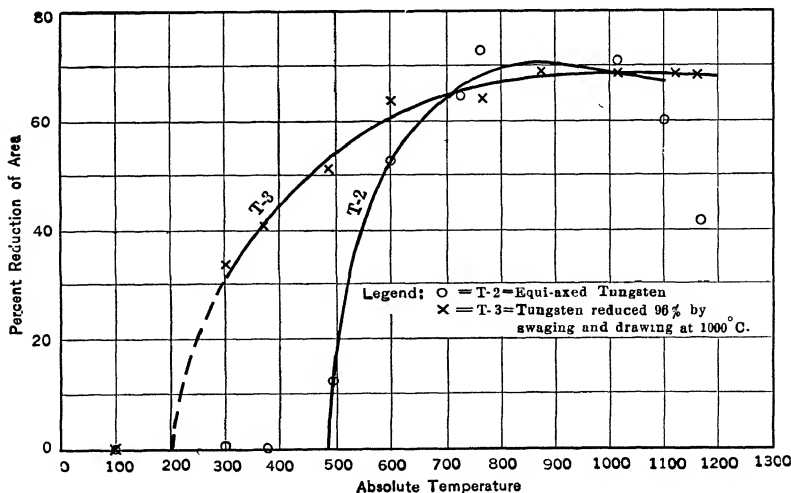


FIG. 59.—PER CENT. REDUCTION OF AREA OF TUNGSTEN WIRES AT VARIOUS TEMPERATURES.

have to be altered. At present it is thought that allotropy is caused by changes in inter-atomic forces or changes in adaptability of positions of the atoms with change in temperature.

Fig. 74 shows that the cohesion of the amorphous phase is about zero at and somewhat below the melting point of the metal, whereas the

cohesion of the crystalline phase assumes a finite value at the melting point. This can be pictured by comparing the cohesion of ice and water at  $0^{\circ}\text{C}.$ ; water has no cohesion and ice has considerable cohesion.

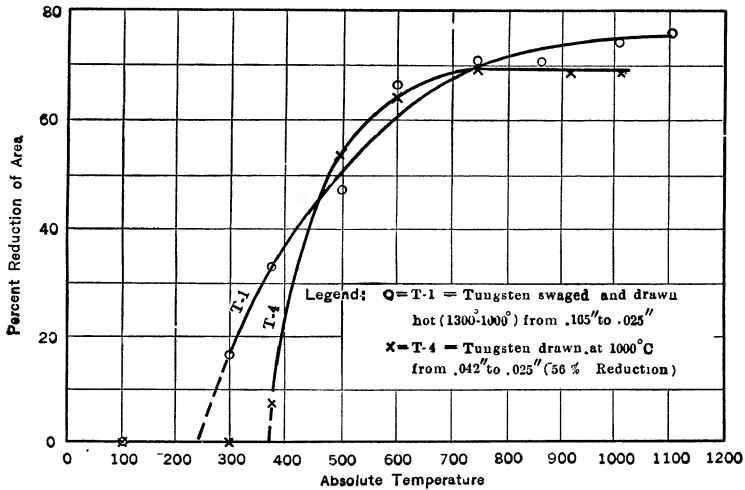


FIG. 60.—REDUCTION OF AREA OF TUNGSTEN WIRES AT VARIOUS TEMPERATURES

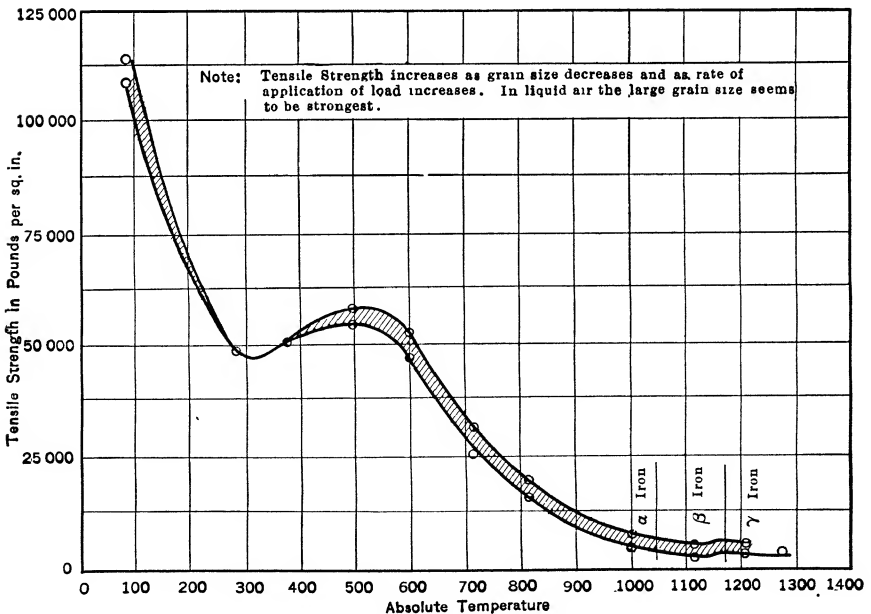


FIG. 61.—TENSILE STRENGTH OF ANNEALED ARMCO IRON AT VARIOUS TEMPERATURES WITH VARIATION IN GRAIN SIZE AND RATE OF APPLICATION OF LOAD.

As the temperature is lowered below the freezing point, the cohesion of the amorphous phase increases at a faster rate than that of the crys-

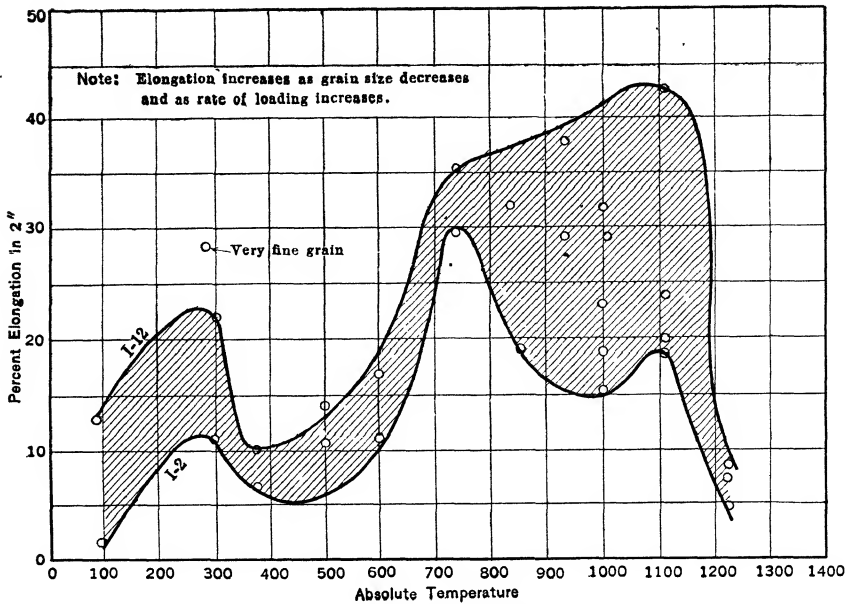


FIG. 62.—PER CENT. ELONGATION OF ANNEALED ARMCO IRON WIRES 0.025 IN. DIAM WITH VARIATION IN GRAIN SIZE AND RATE OF LOADING.

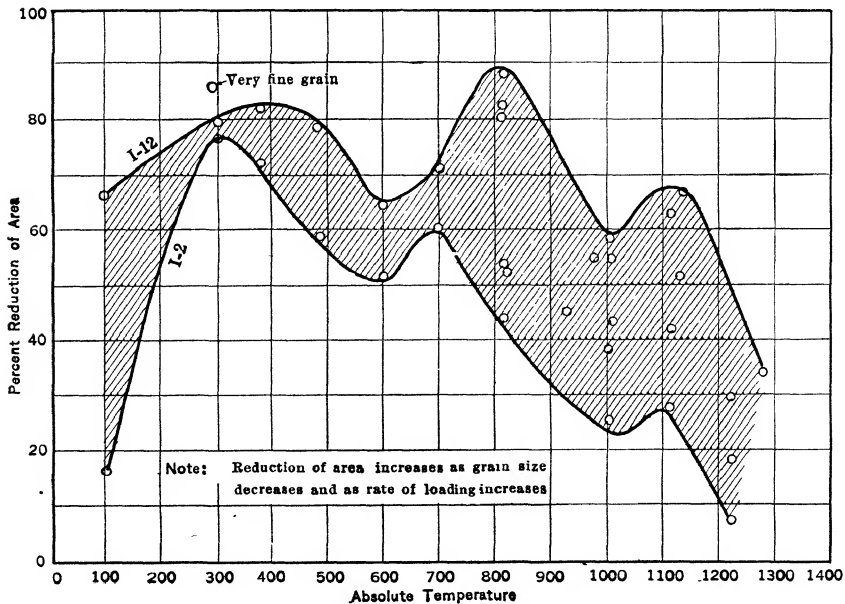


FIG. 63.—PER CENT. REDUCTION OF AREA OF ANNEALED ARMCO IRON WIRES AT VARIOUS TEMPERATURES WITH VARYING GRAIN SIZE AND RATE OF LOADING.

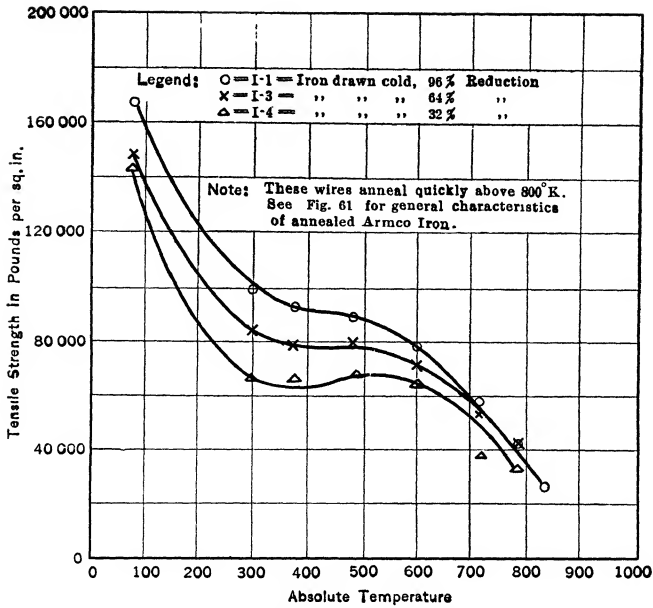


FIG. 64.—TENSILE STRENGTH OF ARMCO WIRES 0.025 IN. DIAM. BELOW ANNEALING TEMPERATURE.

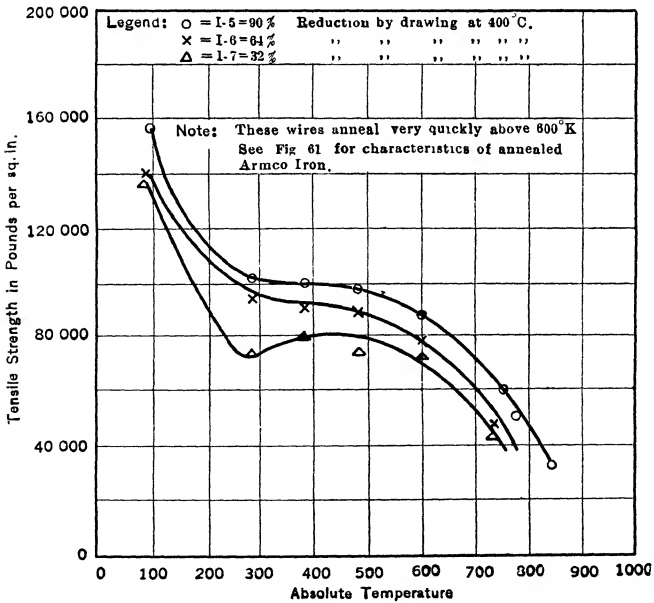


FIG. 65.—TENSILE STRENGTH BELOW ANNEALING TEMPERATURE OF HOT-DRAWN ARMCO IRON WIRES 0.025 IN. DIAM.

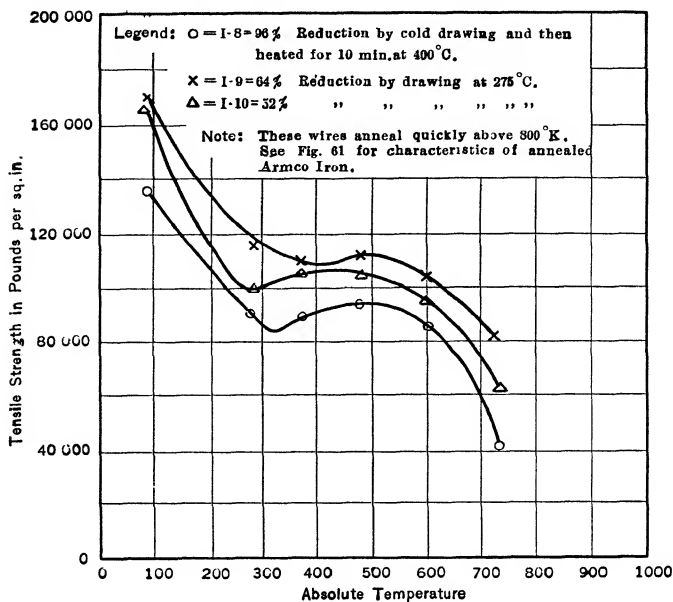


FIG. 66.—TENSILE STRENGTH BELOW ANNEALING TEMPERATURE OF ARMCO IRON WIRES 0.025 IN. DIAM.

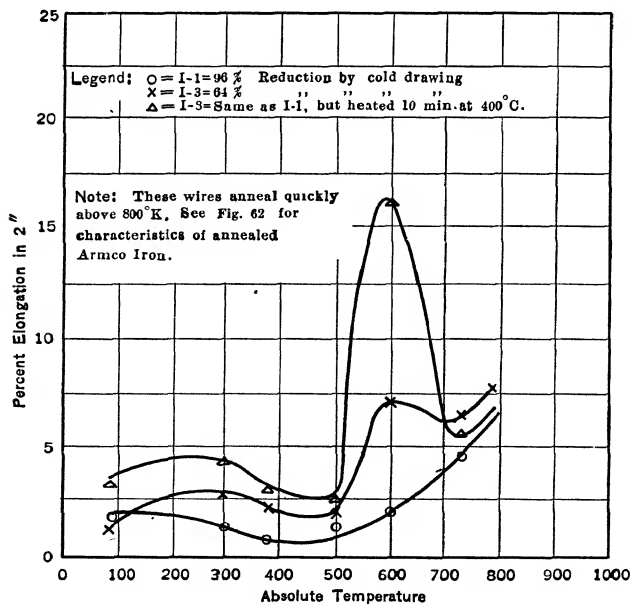


FIG. 67.—PER CENT. ELONGATION BELOW ANNEALING TEMPERATURE OF COLD-DRAWN ARMCO IRON WIRES 0.025 IN. DIAM.

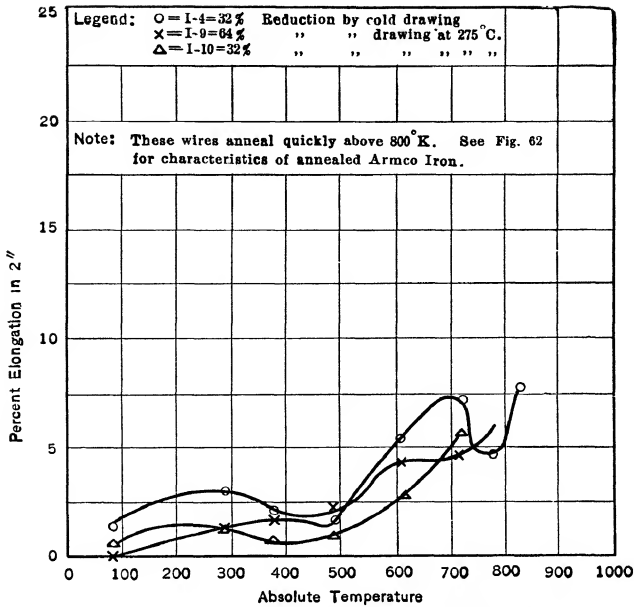


FIG. 68.—PER CENT. ELONGATION BELOW ANNEALING TEMPERATURE OF ARMCO IRON WIRES 0.025 IN. DIAM.

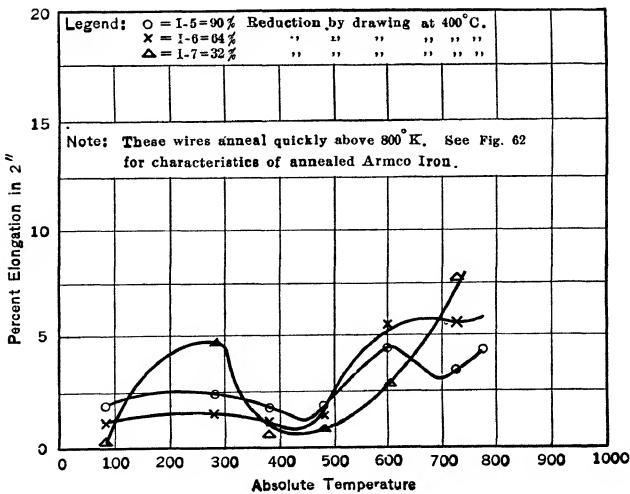


FIG. 69.—PER CENT. ELONGATION BELOW ANNEALING TEMPERATURE OF HOT-DRAWN ARMCO IRON WIRES 0.025 IN. DIAM.

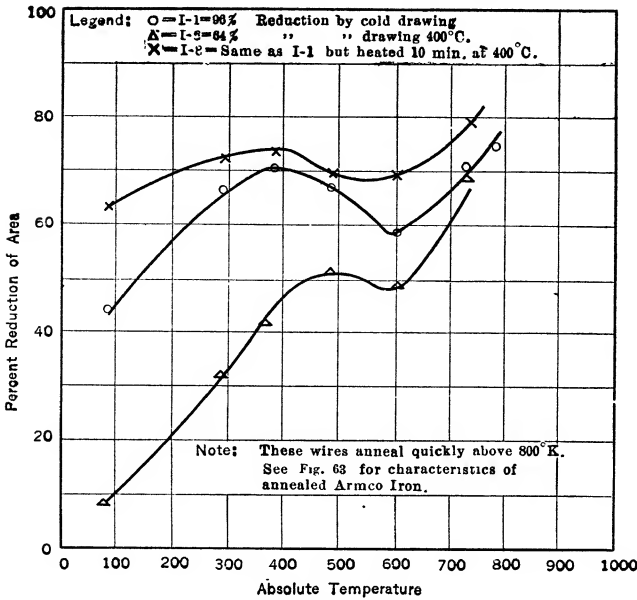


FIG. 70.—PER CENT. REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF ARMCO IRON WIRES 0.025 IN. DIAM.

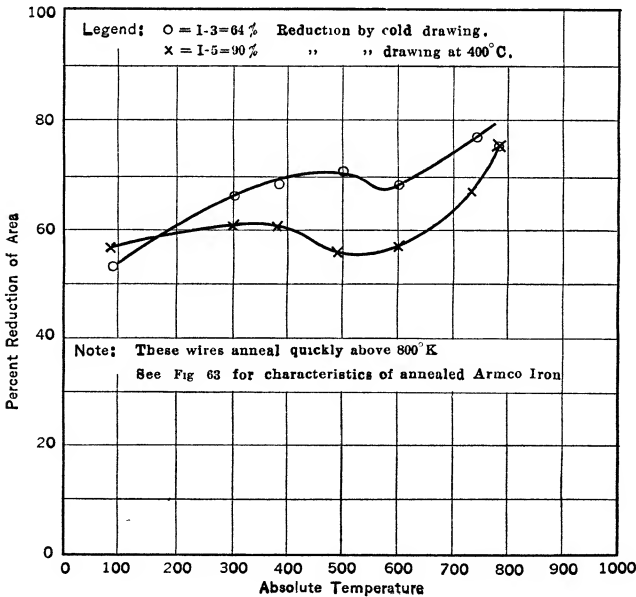


FIG. 71.—PER CENT. REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF ARMCO IRON WIRES 0.025 IN. DIAM.

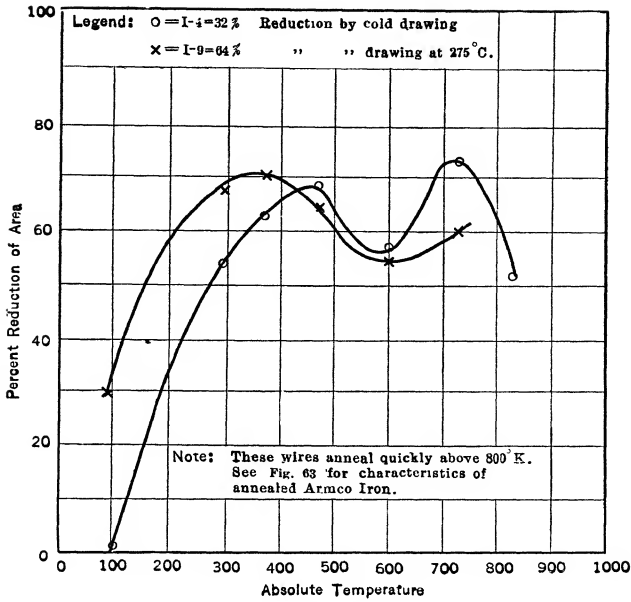


FIG. 72.—PER CENT. REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF ARMCO IRON WIRES 0.025 IN. DIAM.

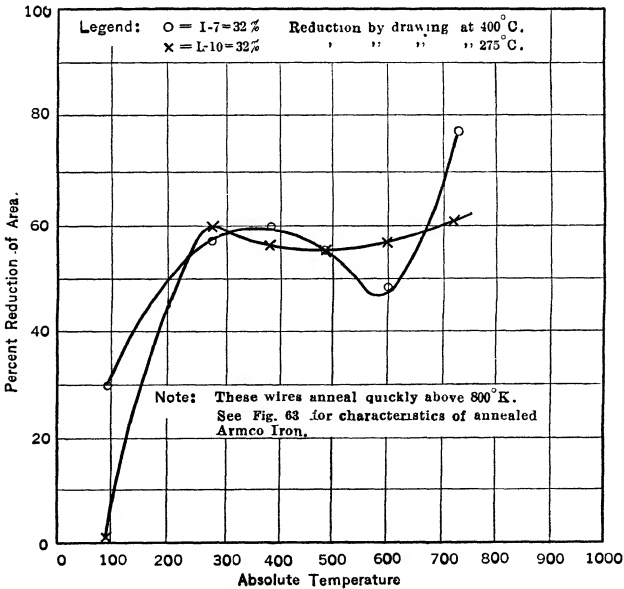


FIG. 73.—PER CENT. REDUCTION OF AREA BELOW ANNEALING TEMPERATURE OF ARMCO IRON WIRES 0.025 IN. DIAM.



talline phase. When the equi-cohesive temperature is reached the cohesion temperature curve of the amorphous phase intersects that of the crystalline. Below the equi-cohesive temperature the cohesion of the amorphous phase is not only greater than that of the crystalline phase but it increases at a faster rate with decrease in temperature. This differential change in cohesion with change in temperature between the amorphous and crystalline phases of a substance is the key to the interpretation of

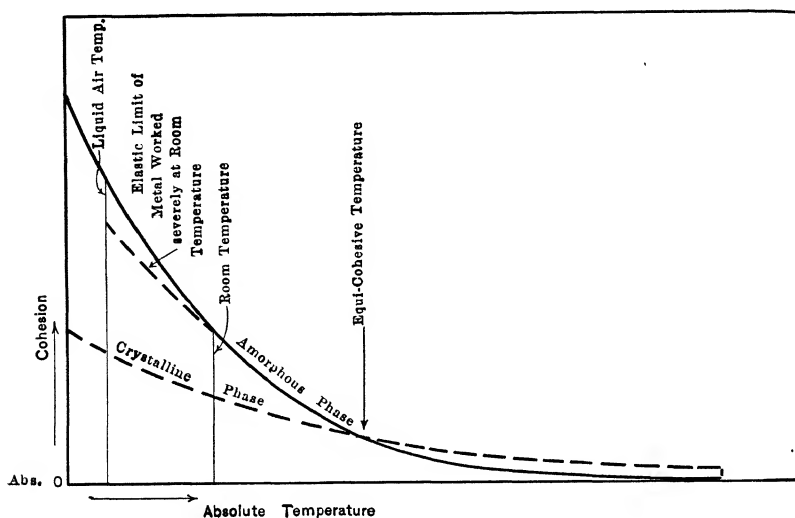


FIG. 74.—DIAGRAM SHOWING CHANGE IN ELASTIC LIMIT AND HENCE DUCTILITY OF A METAL SEVERELY WORKED AT A CERTAIN TEMPERATURE AND TESTED AT AND BELOW THAT TEMPERATURE.

the increase in elongation observed by working a metal at a certain temperature below that of recrystallization and testing at temperatures lower than that at which it was worked.

The cohesion-temperature curves of each substance will have their individual characteristics. They will all be of the same general type, however, as those shown in Fig. 74, with the exception of allotropic substances. Even these will show an intersection of the amorphous and crystalline curves, and a differential change in cohesion at any temperature between the amorphous phase and the stable crystalline allotrope. This knowledge coupled with the information that the amorphous phase at any temperature deforms much more slowly than the crystalline grains at the same temperature enables one to interpret many of the phenomena in connection with the mechanical properties of metals.

#### INTERCRYSTALLINE FRACTURE

It has been demonstrated by Rosenhain and others that at relatively high temperatures the fracture of a metal takes place at the grain bounda-

ries and that at low temperatures fractures take place through the grains themselves. This would indicate that the amorphous phase is weaker than the crystalline at these high temperatures but stronger at low temperature. It should be obvious, however, that the fracture of a metal will take place through the grains at temperatures considerably above that of equal cohesion in a particular metal. This would be true even with very slow loading but the faster the loading the higher will be the temperature at which intercrystalline fracture first appears. The area of the grain boundaries is greater than the cross-section of a test piece on one plane. Intercrystalline fracture, therefore, will not take place when a metal is tested by tension until a temperature is reached at which the cohesion of an area of the amorphous phase equal to the area of fracture along grain boundaries is less than the cohesion of an area of the crystalline material equal to the cross-section of the test piece.

#### TRANS-CRYSTALLINE FRACTURE

When a crystal or a grain is broken in two, it breaks with or without change in shape, that is, deformation. When it breaks without deformation the fracture usually occurs along cleavage planes of the crystal. It is not necessary that any amorphous material be generated by such a break and, in fact, the conditions seem to imply that none can be generated under these conditions. When a crystal of chromium is broken at room temperature, for example, it undergoes no visible deformation. This implies no slipping of one section on another and, so far as we know, the fracture takes place by the cleavage of one group of atoms from another without change of position of any of the atoms relative to the others within the same piece after fracture. If this is the case, no amorphous material could be generated at the point of fracture; if it did generate at the plane where fracture finally took place it should have had greater cohesion at room temperature than the crystalline phase and fracture would not have taken place at that particular plane at that time but some other part of the crystal would have begun to rupture. This process would be kept up until the whole of the crystal had had generated within it amorphous material along thousands of slip planes, but to make this possible the crystal would have to be ductile and it would show permanent change in shape, which is contrary to the conditions observed in chromium.

It seems, therefore, that the property of deformability of a crystal in the solid state depends absolutely and entirely on the ability of that crystal to begin rupture by the slipping of one part on another and the ability of this mechanical slipping action to generate amorphous material which cements the two parts of the crystal together. When a crystal breaks without deformation, it implies the lack of ability to generate

amorphous material along the line of incipient rupture. Similarly, if a load could be applied to any given crystal exactly perpendicular to one of its cleavage planes, and if the load could be maintained uniformly on the whole cross-section of the crystal, would not the fracture take place at the cleavage plane without any deformation and without the generation of any amorphous material? This condition is met to a certain extent when a crystal of a ductile metal is separated into two parts without substantial deformation by means of a hammer blow applied to a cold chisel when the bit of the chisel is placed parallel to a cleavage plane. On the other hand, when the bit is placed non-parallel to a cleavage plane, a hammer blow causes it to cut into the crystal in a manner similar to a chisel cut in ordinary annealed metal. It may be argued that the chisel acts as a wedge and favors rupture a little at a time; this is true but it does not indicate that fracture takes place through amorphous metal which is stronger than crystalline. The argument regarding rupture by degrees holds as well for the crystalline as it does for the amorphous condition, but slow loading favors deformation of amorphous substances at low temperature.

Non-metallic crystals, like calcite and fluorite, break easily at room temperature and fracture takes place along the cleavage planes. If we assume that amorphous material is generated at the surface of fracture in a brittle metallic crystal, we should make the same assumption for non-metallic crystals; this seems hardly necessary with our present knowledge. But if added evidence is needed, it is found cogently in such brittle metals as coarse-grained bismuth and zinc. In these metals fracture always avoids the grain boundaries, which are filled with the amorphous material. Would not fracture also avoid an intra-crystalline amorphous plane? The foregoing assumes a condition of no internal stresses between amorphous and crystalline metal.

The generation of amorphous metal at the slip planes implies malleability and ductility in a metal. As the load is applied to the crystal, no permanent deformation takes place until the elastic limit is exceeded, but the atoms change their positions as a result of the load and permanent deformation would first take place where the atoms were strained the most.

Assuming that the direction of the load on the crystal is such that it is not perpendicular to a cleavage plane, the first incipient rupture of the crystal would not be perpendicular to the direction of the load. Slippage on each side of the incipient rupture must then take place but in the act of slipping some of the atoms that have already been stressed to the point of separation from one another (especially in the crystalline arrangement) are actually mechanically decrystallized, and hence amor- phized. The amorphization is probably not complete until the two parts have slipped a distance, which may be considerable when compared

to the diameter of an atom. The mobile state of the material at the slip plane probably occurs while the amorphous material is in the process of formation. A locally high temperature is no doubt also developed at the slip plane, due to the mechanical action. The amorphous metal is more mobile at the high temperatures than after cooling. As soon as the amorphous phase is completely formed, the friction probably decreases and the heat previously generated by the mechanical action is dissipated into the crystalline parts and the amorphous phase cools and becomes rigid. Immediately after formation the amorphous slip planes are probably in a high state of internal stress, which is relieved gradually. This gradual release probably accounts for the change in properties of a metal with age. At the same time the slipping on this particular section removes the local stress and transfers it to another section. The process of amorphization will then take place at the other planes of incipient rupture. In this manner the process of deformation of a crystalline material at low temperatures automatically strengthens the parts deformed and forces any further deformation to take place in the other crystalline parts. Immediately after deformation, probably both amorphous and crystalline metal are in a state of internal stress, the direction being in general the same as that of deformation. The removal of the stresses in the amorphous phase with age probably also removes the stresses in the crystalline metal. It is conceivable that this readjustment may require infinite time at very low temperatures and a matter of seconds at high temperatures. At intermediate temperatures we should then have all gradations for the release of internal stresses from a few minutes or seconds to infinite time.

We all know that the resistance to deformation of pure iron (ferrite) can be increased in the neighborhood of three times by laminating it with 13 per cent. of the hard and brittle material, cementite, to form pearlite. The ferrite is strengthened in this manner by the reinforcement of the cementite. When a load is applied to pearlite, it is the ferrite which undergoes deformation, but it does not deform before a load is reached equal to three times the load at which ferrite alone will deform. This result is produced by reinforcing a deformable metal with one that is absolutely brittle. Similarly, as the deformation of a crystal proceeds and as the crystalline phase becomes more and more reinforced by the amorphous metal, a condition finally obtains in which the load necessary to further deform the crystalline phase with its reinforcement is equal to the breaking load of the amorphous phase at the slip planes. Rupture finally takes place largely along the amorphous slip planes, either those formed by previous deformation or those formed at the time of rupture. This is the process of trans-crystalline fracture of a single ductile grain.

After rupture there will be many grain fragments within which the

atoms have the same relative positions as before deformation. If these could be isolated they could be deformed. A grain or crystal that has been broken in tension can be subjected to further deformation by compression as in rolling. The limit of deformation of a crystalline substance is therefore not reached when a metal is broken in tension. A good example of this is found in the wire-drawing of a soft metal like copper. A piece of copper which shows only 40 per cent. elongation in 2 in. in a tensile test may have its length increased 10,000 or more times without intermediate annealing, by rolling and wire-drawing. So far as is now known, no crystalline material has ever been deformed mechanically as much as it is possible to deform it. The limit of deformation would theoretically be reached only when every atom had been transformed into the amorphous condition. It is not possible to do that because the deformation load must be transmitted to the crystalline metal largely through amorphous material. After extreme industrial deformations, it is probable that at least 90 per cent. of the material is still in the crystalline form.

It is generally admitted that intercrystalline fracture takes place in the amorphous material or at the boundaries between amorphous and crystalline material. I shall also take the position that trans-crystalline fracture takes place either at the amorphous slip planes or at the cleavage planes of the grains without generation of amorphous metal.

The crystalline material in metals may be divided into two general classes: easily deformable and brittle. A given crystal might be in one class at one temperature and in the other class at another. Large zinc grains would be in the first class at 150° C. and in the second class at room temperature. When coarse-grained cast zinc is broken at ordinary temperature the fracture shows grains, which reflect light directionally. This same surface could be polished and the grains would be entirely invisible. Beilby has taught us that the polishing produces a thin layer of amorphous zinc on the surface which has no directional properties and hence the whole surface has a uniform tint. The fact that the grains on the fracture show directional reflection indicates that there is no amorphous metal on the surfaces of the broken grains or that at least it is so thin that light passes through it. I am of the opinion that the fracture has taken place on cleavage planes without the generation of amorphous metal. Chromium, antimony, and bismuth, as well as other brittle metals, act in a similar manner when fractured cold. These metals would be in the second class.

When coarse-grained cast tin is broken in tension at ordinary temperature the fracture gives no indication of the grain boundaries. The grains are easily deformable and would be in the first class. If we polish a cross-section of the tin near the fracture and etch it, the grains are easily visible. Why are the tin grains not visible after fracture? The

simple explanation would be that they are coated with amorphous metal. Both surfaces of the fracture are similar, so it is evident that rupture took place in amorphous metal or the rubbing of the two parts together after rupture produced the amorphous coating. The fracture surface of coarse-grained tin suggests that rupture has taken place largely by shearing action. The same is true of many ductile metals.

Other metals rupture partly at the cleavage planes of the grains without generation of amorphous metal and partly at slip planes after local deformation and hence at the amorphous planes.

### RUPTURE

The breaking load of a single constituent ductile metal when tested in tension is controlled by the cohesion of the amorphous phase but is not equal to it. The cohesion of the amorphous phase will always be different from that indicated by the breaking load because all parts of the cross-section do not rupture simultaneously and because the total area of rupture varies on cross-sections of equal size due to causes such as differences in grain size and degree of cold work. The property of ductility in a metal seems to favor rupture along a long path but at the same time promotes rupture by degrees. The rupture of sample T-2 at room temperature and at 100° C. was not accompanied by deformation. It took place over the whole area of fracture within a very short time period but, due to lack of perfect homogeneity, some parts must have given way an instant before final rupture occurred. If rupture took place at every point at the same instant we would have data for the measurement of absolute cohesion, which no one has been able to obtain to date. The rupture of tungsten in its brittle range, however, is more nearly simultaneous at all points of the fracture than at higher temperatures in the ductile range. Ductility seems to favor non-homogeneity and this favors rupture by degrees, which in turn makes the breaking load low in proportion to the absolute cohesion of the amorphous phase.

Rupture by degrees in a ductile metal can be studied nicely with the Kron springless dial scale used in the experiments above 200° C. After the maximum load had been reached, the dial would first move slowly in the direction of decreasing load and then more rapidly until a load about one-half or two-thirds the maximum was recorded, after which the wire would break and the dial would spring quickly to its zero point. The time during which rupture was taking place was considerable, often as much as 5 sec. The foregoing discussion should be kept in mind when interpreting the expression "the breaking load is controlled by the amorphous phase."

Many facts indicate that the amorphous phase controls the breaking load. Annealed copper, C-2 for example, had a tensile strength of 31,150 lb. per sq. in. (21.8 kg. per sq. mm.) at room temperature. The break-

ing load gradually increased to 67,400 lb. per sq. in. (47.18 kg. per sq. mm.) as the deformation progressed to 96 per cent. reduction by cold drawing. The principal difference between the annealed and the hard-drawn copper was in the quantity and arrangement of the amorphous phase and as its quantity increased the breaking load increased. If the hard-drawn wire is probably more than 90 per cent. crystalline, why does not fracture take place through the crystalline material with slight breaking load? I believe it is because the amorphous phase is primarily the continuous one and the load must be transmitted to the crystalline phase through it; and the crystalline phase cannot commence rupture without generating new amorphous metal. This process continues until rupture occurs in or at the amorphous planes.

We might assume that the surface of the fracture of the annealed copper is entirely amorphous as is also that of the hard-drawn wire. The reason then that the latter has a higher breaking load is largely due to a more intricate path of rupture. The intricacy of the path of rupture would naturally be greater as the wire drawing became more severe. This accords with the idea that the elastic limit increases with the intricacy of the path of rupture and hence with the quantity of the amorphous phase. The hard-drawn wire may also be more homogeneous and this would produce a more simultaneous rupture of the various parts of the cross-section than in annealed wire.

The directional properties of the crystalline phase force the formation of amorphous phase along intricate paths and hence the breaking load of the complex structure is probably much greater than that of the stronger (amorphous) phase itself.<sup>11</sup>

#### EXPLANATION OF BEILBY'S RESULTS

The reason that hard-drawn gold, silver, and copper wires showed less than 1 per cent. elongation when tested at room temperature is given in the preceding. The resistance to deformation in the crystalline phase had been increased by the amorphous reinforcement planes until it was about the same as the resistance to rupture by tension of the amorphous material itself. The wires therefore broke at room temperature with very little elongation. On cooling to the temperature of liquid air, the resistance of the amorphous phase to rupture increased at a more

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<sup>11</sup> This idea of a combination of amorphous and crystalline metals, with higher cohesion than either singly, strongly suggests a mechanical explanation for the cohesion of solid solutions. The atoms of the different components having different atomic volumes would cause an intricate atomic path of rupture which would increase the cohesion. This is suggested quantitatively by the fact that solid solutions showing the highest increase in cohesion usually consist of atoms having great variation in atomic volume. The martensitic state in steel also strongly suggests a combination of two physically different constituents with higher cohesion than either one.

rapid rate than the resistance to deformation of the crystalline phase. Since the resistance to deformation of a structure composed of fragments of crystalline material reinforced with thin sheets of amorphous material would be governed partly by the cohesion of each phase, the final resistance to deformation at liquid-air temperatures would be represented by a weighted average of the increase in cohesion of each phase. It must be less than the increase in cohesion of the amorphous phase and greater than the increase in cohesion of the crystalline phase. The resistance to deformation of the deformed grains taken as a whole must therefore be less at liquid-air temperatures than the resistance to rupture of the amorphous phase but still greater than the resistance to deformation of the crystalline phase unreinforced (not strain hardened).

The resistance to deformation of the grains taken as a whole controls the elastic limit. The elastic limit at liquid-air temperatures must therefore be less than the breaking load of the amorphous phase, and hence the wires were ductile. At room temperature the elastic limits were substantially equal to the breaking loads of the hard-drawn wires but considerably less than the breaking loads of the same wires at the temperature of liquid air. The elastic limits of these wires were greater, however, in liquid air than at room temperature, but the rate of increase of elastic limit with decrease in temperature is not as rapid as the rate of increase in the strength of the amorphous phase. This explanation is shown diagrammatically in Fig. 74. This diagram shows a condition in which the elastic limit is equal to the breaking load at room temperature. If the wires had been drawn at room temperature to such an extent that their elastic limits were considerably less than their breaking loads, the elastic-limit curve would approach the breaking-load curve at a temperature higher than room temperature.

#### MECHANICAL PROPERTIES OF COPPER WIRES

*Elongation.*—The results of the tests on copper given in Tables 1 to 9, inclusive, and in Figs. 43 to 55, inclusive, confirm these general ideas. The hard-drawn copper wire C-1, which had an elongation of only 0.75 per cent. at room temperature, had an elongation of 6.25 per cent. in liquid air. The elongation is almost nil at 200° C. At 330° C. it anneals quickly and its elongation will then depend on other factors. Similarly, sample C-3 shows a gradual increase in elongation as the temperature is lowered below that of deformation and a gradual decrease as the temperature is increased until the first annealing takes place. This is also shown in samples C-4, C-5, C-6, C-7, C-8, and C-9, some of which were worked at room temperature and some at higher temperatures. The decrease in elongation with increase in temperature above that of working should be due to the differential change in cohesion between the amorphous and crystalline phases.



Let us consider sample C-4, for example, which had an elongation of 2.12 per cent. at room temperature, after reduction of 32 per cent. by cold drawing. At 200° C. its elongation was only 0.75 per cent. Why did its elongation decrease with increase in temperature? The resistance to deformation of the deformed crystalline grains had been so increased at room temperature by the generation of amorphous metal at the slip planes that only a little more deformation was possible by tensile load before the sample would break. On increasing the temperature the resistance to deformation will decrease but the resistance to rupture in the amorphous phase, which controls the breaking load, will decrease at a still faster rate with increase in temperature. The amorphous reinforcement planes within the strain-hardened grains will also be less effective for reinforcement. This would tend to decrease the resistance to deformation of the deformed crystalline grains in proportion, but not equal to the decrease in cohesion of the amorphous phase. The total decrease in resistance to deformation with increase in temperature will be a weighted average between the decrease in the cohesion of the crystalline phase and that of the amorphous phase at the slip planes. The decrease in breaking load, however, will be directly proportional to the decrease in cohesion of the amorphous phase, which in turn must be greater than the decrease in resistance to deformation of the deformed grains. This is a general rule applicable to all metals within the proper temperature range.

Sample C-2 measured 485 grains per square millimeter after annealing. Its elongation decreases from the lowest temperature, -190° C., to the highest temperature tested, 950° C. Its elongation at the lower temperatures is less than ordinary annealed copper because it has comparatively few grains on a cross-section. A finer grained sample was obtained by using a lower annealing temperature and this was tested at room temperature. Refer to C-10, shown in Table 2. The measurements showed 6200 grains per square millimeter. The tensile strength, elongation, and reduction of area were all increased by a decrease in grain size. The elongation of annealed copper at all temperatures was less the larger the grain size. Compare C-1-a-450, Table 1, with C-2-a-450, Table 2; the former is annealed but has a small grain size because the wire has been annealed during test and the temperature is rather low and there has not been time for marked growth of grain. Its elongation is about twice that of the coarser grained (C-2) sample. Sample C-3-b-450, Table 3, is also annealed, but coarser grained than C-1-b-450 because its deformation before test was less.

In general, the elongation with any given grain size decreases with increase in temperature. It may be that a fine-grained sample will have a greater elongation at a certain temperature than a coarse-grained sample at a certain lower temperature but, in general, the elongation of

each sample will decrease with rising temperature; this condition is shown in Fig. 44.

Mr. W. H. Bassett has called attention to the fact that the elongation of annealed copper is greater after an anneal at 500° to 600° C. than after either lower or higher anneals, and that a maximum elongation at a given temperature might be expected after an anneal higher than that of C-10 and lower than that of C-2. This information is confirmed by results of Mathewson and Thalheimer. From the writer's experiments, it seems that this relation does not hold at higher temperatures, but that reduction in grain size favors both high elongation and high reduction of area.

The elongation will also increase as the rate of applying the load increases. Very rapid application of the load, especially at temperatures between 450° C and 950° C., may double the elongation obtained by slow loading. This is in keeping with the general proposition that the amorphous phase deforms more slowly than the crystalline phase with a given absolute cohesion. The absolute cohesion of the amorphous phase is less than that of the crystalline phase in the temperature region in which the rapid loading was used, but the relative cohesion of the two phases with short duration of the load may be greatly changed by changing the rate of loading. If, for instance, a hammer blow is struck on a piece of pitch, only a slight dent may result or the pitch may break into pieces. If the hammer, however, is placed on top of the piece of pitch and left there over night or for a few days, it may actually bury itself, and at any rate it will leave an impression. This is characteristic of amorphous substances, especially in their temperature regions where flow takes place. If a load of  $x$  lb. is required to break a piece of glass at a red heat in 10 min., a load of  $2x$  lb. will not break the glass immediately but will do so in less than 10 min. The absolute cohesion of the particular piece of glass would be considered as the load per unit of cross-section that would break it in an infinite time. The difference between the absolute cohesion and the observed cohesion by breaking it in any ordinary short-time period is entirely a function of the rate of loading; the crystalline phase is not so much affected by the rate of loading, and hence rapid loading has the effect of increasing the cohesion of the amorphous phase more than that of the crystalline.

It may be that some of the copper wires have spontaneously annealed during test in the temperature region permitting grain growth. It is certain that the cold-worked wires annealed at the higher temperatures but when these were deformed again during the test another annealing may have taken place. If this has not happened in the particular wires tested in these experiments, it may happen, and this would vary the elongation. After a wire has stretched, say 10 per cent. of its original length, recrystallization may take place and then the test proceeds on a sample

with a structure similar to that at the beginning of the test. This may account for some of the erratic elongation measurements obtained by various experimenters at temperatures in the grain-growth region.

*Tensile Strength of Copper Wire.*—The tensile strength of all copper wires decreases from liquid-air temperatures to the highest temperature tested. Some of the curves show an apparent discontinuity between 200° C. and 330° C. These wires were loaded rapidly to avoid annealing. The wires that were held at 330° C. for 10 min. or more before test were annealed. The annealed copper itself, C-2, shows the normal decrease in tensile strength between 200° C. and 330° C. This is about the same as was observed by Bengough<sup>12</sup> and Le Chatelier.<sup>13</sup>

The tensile strength increases as the grain size decreases. This is especially marked below the recrystallization temperature and not marked above. The tensile strength also increases at the higher temperatures as the rate of loading increases. This is in keeping with the general proposition that the amorphous phase has the ability to withstand for a short time without rupture loads that would break it in a longer time. The apparent temperature-cohesion curves of the amorphous and crystalline phases may be raised several hundred degrees above that of the equilibrium positions by rapid loading. Sample C-1-a-950, for example, had the same breaking load when broken in 5 sec. as sample C-1-b-750, which was broken in 15 min. The fast rate of loading in this instance has produced the same increase in cohesion as would be produced by lowering the temperature 200° C.

The tensile strength of the cold-worked copper wires decreases gradually from liquid-air temperatures to 330° C. The difference in tensile strength at 330° C. between the recrystallized and unrecrystallized samples is very marked. The decrease in sample C-1 was 39 per cent. The difference in strength at 330° C. between some of the strain-hardened samples and the annealed sample, C-2, is about as 2 is to 1. If the hypothesis is correct, that the breaking load depends on the cohesion of the amorphous phase, any variation in breaking load of a given metal at any temperature will depend on the area of the path of rupture through the amorphous phase and on the area that ruptures at any given instant. Since the variations in reduction of area between annealed and cold-worked copper were slight, we may assume that the area of the path of rupture through the amorphous phase of cold-worked copper is substantially twice that of annealed copper. This ratio is nearly constant from liquid-air temperatures to the annealing point of copper. Cold work increases the area of rupture through the amorphous phase; slight cold

<sup>12</sup> *Op. cit.*

<sup>13</sup> Le Chatelier's tenacity-temperature curve of copper is reproduced on p. 143 of Bengough's paper.

work increases this path but little and severe cold work increases it to a maximum.

The process of recrystallization is one that shortens the path of rupture along amorphous planes and at the same time substitutes a normal equiaxed grain structure. The individual equiaxed grains, however, have less resistance to deformation than the resistance to rupture at their boundaries, and these grains cannot deform without generating within them amorphous material that increases the area of amorphous metal on any given cross-section as the deformation proceeds. Not only, therefore, is recrystallization coincident with a marked decrease in the breaking load but it produces a marked increase in the ductility.

*Reduction of Area of Copper Wires.*—In general, the reduction of area of both annealed and cold-worked copper wires increases from liquid-air temperatures to about 330° C., after which a marked decrease takes place with normal loading. The reduction of area increased in the annealed samples as the grain size decreased. It is, in general, greater in the annealed wires than in the cold-worked wires but the condition may obtain in which the reverse is true. The reduction of area is not only dependent on the grain size but also on the number of grains, whether strain-hardened or not, exposed on the whole cross-section of the wire. In general, the more grains exposed on the cross-section, the greater will be the reduction of area. If, for example, a wire contains 1000 equiaxed grains on a cross-section, it will have a greater reduction of area than one containing 1000 strain-hardened grains on its cross-section. On the other hand, if a wire contains only 100 annealed grains on its cross-section, its reduction of area may be less than that of a wire of the same size containing several thousand strain-hardened grains on its cross-section. From the results of Mathewson and Thalheimer,<sup>14</sup> it would appear that the reduction of area of annealed copper at room temperature is a maximum at a certain intermediate grain size produced after an anneal at about 600° to 700° C. Their micrographs, however, indicate that the grain size after 600° anneal is but very little different from that after a 400° or 500° anneal, and the percent reduction of area shows a correspondingly slight variation. On the contrary, as soon as the grain size begins to increase markedly, namely at and above 800° C., the reduction of area as well as the elongation begin to fall rapidly. It would not be surprising if all metals at any given temperature would have maximum elongation and reduction of area with a certain small grain size, and either smaller or larger grains might produce less elongation or less reduction of area. On the contrary, the tensile strength should always increase with decrease in grain size.

The reduction of area seems to depend on the ability of a wire to

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<sup>14</sup> *Trans.* (1916) 55, 467.

break by degrees. A brittle metal has no reduction of area; a ductile metal always has reduction of area. If the mechanism of rupture of a ductile wire permits the rupture of one interior part while adjacent parts remain intact, these may draw together and fill up the opening left by the ruptured part. In proportion to the ability of a metal to break in this manner, the reduction of area increases.

Above 330° C. the reduction of area depends to a marked extent on the rate of loading. Rapid loading increases the reduction of area and slow loading decreases it. At the higher temperatures slow loading seems to produce intercrystalline fracture but fast loading produces trans-crystalline fracture. This would be expected when the variation in cohesion of the amorphous phase with change in rate of loading is considered. Very rapid loading at temperatures between 600° C. and 950° C. produces almost 100 per cent. reduction in area. The reduction of area of sample C-1-a-750 was 99.5 per cent. This is somewhat analogous to glass, which is an amorphous material, and strongly suggests the existence of amorphous material in copper. When glass at the proper red heat is broken in tension, its reduction of area is about 100 per cent. The making of mineral wool depends on this property. If the glass is loaded rapidly a small globule can be formed into a thread many feet in length. The copper acts in a manner somewhat analogous to glass, but of course a complete analogy does not exist because of the preponderance of crystalline material present.

### MECHANICAL PROPERTIES OF TUNGSTEN WIRES

*Elongation of Tungsten Wires.*—Recrystallized tungsten is brittle at 100° C. and below. Its fracture at these low temperatures is at the grain boundaries, that is, intercrystalline. At 200° C., the average elongation in 2 in. is 8.85 per cent. and the fracture is partly trans- and partly intercrystalline. At 330° C., the average elongation is 21.8 per cent. and the fracture is entirely trans-crystalline. The elongation above 330° C. decreases somewhat with increase in temperature but the normal decrease is not properly shown by the figures given in Table 11. This equiaxed tungsten wire was recrystallized in short lengths and microscopic examination showed that near the surface some of the wires had large grains caused by germination. These large grains tend to decrease the elongation, which accounts for the variations observed in this property.

No tensile tests on tungsten have been made at temperatures near its melting point but there is every reason to believe that the fracture near the melting point would be intercrystalline, as in other metals. The fracture at and below 100° C. is also intercrystalline. The reasons for the intercrystalline fracture at high and low temperatures, however, are very different. At high temperatures, intercrystalline fracture

takes place because of the weakness of the amorphous phase between the grains. I believe that intercrystalline fracture takes place at low temperatures because of the difference in coefficient of expansion between the amorphous and crystalline phases. It is probable that the coefficient of expansion of the amorphous phase is greater at high temperatures than that of the crystalline phase, and that the opposite is true at low temperatures. There should be an intermediate temperature at which the coefficient of expansion of the two phases is equal and they will be nearly the same for a considerable range in temperature above and below that of equal expansion. As the temperature decreases below that of equal expansion, there will be a differential coefficient of expansion, that of the amorphous phase varying at a more rapid rate than that of the crystalline phase. At high temperatures, any difference in coefficient of expansion between the two phases will be compensated for by the flow in the amorphous phase. At low temperatures, the difference in the coefficient of expansion between the two phases will not only be great, but both phases are comparatively rigid and any difference in expansion must manifest itself by internal strain. This internal strain will be at the junction between the amorphous and crystalline phases, so that when external load is applied rupture will take place before a load can be applied equal to the elastic limit of the crystalline phase.

I had developed this hypothesis before reading Prof. T. W. Richards' paper, "The Present Aspect of the Hypothesis of Compressible Atoms."<sup>15</sup> After the cohesion-temperature curves were known, this variation in coefficient of expansion could have been predicted from Prof. Richards' conclusion that this property varies inversely as the cohesion. I arrived at these conclusions regarding the expansion of the amorphous and crystalline phases from the study of the expansion laws of liquids and amorphous solids such as wax and by comparing these with crystalline materials. In some cases the expansion curves of liquid and solid metals were available. A good example of a non-metallic material is found in  $\text{SiO}_2$ . Comparisons were made between quartz and fused silica.

The grains in sample T-2, as shown in Fig. 24, are not exactly equiaxed; they are longer in the direction of drawing than in a transverse direction. This makes the area of rupture through the amorphous phase greater than if the grains had been exactly equiaxed. This wire, therefore, assumes ductility on heating at a lower temperature than if the grains had been exactly equiaxed.

As the temperature decreases, the cohesion of both amorphous and crystalline phases increases according to the general type of curves shown in Fig. 74. The cohesion of an aggregate of recrystallized grains reaches a maximum at about room temperature, below which it decreases.

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<sup>15</sup> *Journal*, American Chemical Society (1914) **36**, 2417-2439.

T-2, for example, was only about one-third as strong in liquid air as at room temperature. This is consistent with the idea that the difference in coefficient of expansion of the two phases causes internal strains.

An isolated grain of tungsten is somewhat malleable at room temperature. It is only when these malleable grains are cemented together by the amorphous metal that the aggregate is brittle at room temperature. As the temperature increases above room temperature, the internal stresses become less until a point is reached at which the intercrystalline breaking load is greater than the resistance to deformation of the crystalline phase. The metal then becomes ductile. The resistance to intercrystalline fracture will not reach that of the deformation of the crystalline phase at the same temperature in each individual grain. The change from complete brittleness to complete ductility, therefore, takes place through a small temperature range; this range does not exceed and may be less than  $120^{\circ}\text{C}$ .

The elongation of the tungsten wires that were worked below the recrystallization temperature is of interest and importance because it has been found possible in this manner to produce tungsten wires that are ductile at room temperature. Sample T-1, for instance, has an elongation of 3.15 per cent. at room temperature; this wire can be bent cold or can be drawn to a certain extent cold. The elongation of tungsten wires is shown in Fig. 58. The worked wires are all brittle in liquid air, but T-1 and T-3 are ductile at room temperature. T-4 is brittle at room temperature but assumes slight ductility at  $100^{\circ}\text{C}$ . and maximum ductility at  $216^{\circ}\text{C}$ . The maximum elongation of T-1 is at  $100^{\circ}\text{C}$ . and of T-3 at  $100^{\circ}\text{C}$ . Above the maximum the elongations decrease in a regular manner with increase in temperature. This decrease in elongation is the result of the same laws that have been discussed for copper. The decrease in elongation below the maximum, however, is caused by the internal strains due to the difference in coefficient of expansion between the amorphous and crystalline phases. The reason that the worked tungsten wires retain ductility at lower temperatures than the recrystallized metal is that the path of rupture along the grain boundaries of the worked tungsten is so great that rupture is forced to take place through the deformed grains or fibers themselves. The amorphous metal generated within the deformed grains also forms a long path of rupture, which in turn requires a large breaking load. In addition to this, the amorphous metal generated within the deformed grains may not be in such a state of strain as the amorphous metal at the grain boundaries of annealed metal; this amorphous metal will have been generated at a comparatively low temperature. If any stress had existed between it and the crystalline metal immediately after deformation, it would seem to have been in a direction that the differential coefficient of expansion would relieve. This is suggested by the observed fact that if the tem-

perature at which tungsten is deformed is high, brittleness develops on cooling at a higher temperature than if the temperature of deformation has been low. The large breaking load, therefore, will force the crystal-line fragments of the deformed grains to further deform before rupture can take place, that is, the metal becomes ductile. It will be noted that the worked tungsten wires are more ductile at certain low temperatures than the equiaxed wires but that the maximum ductility of the latter is greater than that of the former. The maximum elongation of tungsten wires is lower as the deformation increases. In general, the maximum elongation of a worked tungsten wire occurs at a lower temperature as the degree of deformation increases and as the temperature of deformation decreases. It was found possible to put a slight permanent bend in a fibrous tungsten wire, 0.031 in. (0.0254 mm.) diameter, in liquid air.

*Tensile Strength of Tungsten Wires.*—Equiaxed tungsten, as shown in Table 11, has a tensile strength of only about 60,000 lb. per sq. in. (42.16 kg. per sq. mm.) at  $-190^{\circ}\text{C}$ . At room temperature the strength is increased to 168,000 lb. per sq. in. (118.05 kg. per sq. mm.). Between room temperature and  $200^{\circ}\text{C}$ . the decrease in tensile strength is great. The tensile strength at  $100^{\circ}\text{C}$ . is 50,000 lb. per sq. in. (35.13 kg. per sq. mm.) greater than at  $220^{\circ}\text{C}$ . This is the region in which the metal changes from a brittle to a ductile condition. Above  $200^{\circ}\text{C}$ ., which is entirely within the ductile region, the decrease in tensile strength is gradual with increase in temperature. The same marked increase in the strength of iron is noted between room and liquid-air temperatures. These results suggest that rupture takes place by degrees (a little at a time) in ductile metals and more nearly simultaneously at all parts of the cross-section in brittle metals.

All types of recrystallized tungsten wires do not have the strength properties shown in Table 11. The strength properties will depend on the grain size and the shapes of the grains. Grains that are exactly equiaxed produce weaker metal than is shown in Table 11. All of the worked tungsten wires show less tensile strength at liquid-air temperature than at room temperature. It is thought that this variation is due to the difference in coefficient of expansion between the amorphous and crystalline phases. Above room temperature the tensile strength decreases gradually with increase in temperature. The highest temperature used in the tests was  $900^{\circ}\text{C}$ ., which is about  $500^{\circ}\text{C}$ . below recrystallization.

#### SPECIAL TESTS ON TUNGSTEN

Table 14 gives the results of some special tests on the recrystallized tungsten wires. It had been shown that these wires had an average breaking load of about 168,000 lb. per sq. in. (118.05 kg. per sq. mm.)



at room temperature and only about 60,000 lb. per sq. in. (42.16 kg. per sq. mm.) in liquid air. It seemed probable that if one of these wires could be stressed more than 60,000 lb. per sq. in. at room temperature and immersed in liquid air while under stress it might break spontaneously. Consequently a wire was stressed to 101,000 lb. per sq. in. (71.00 kg. per sq. mm.) at room temperature and was immersed in liquid air under stress, but it did not break. The load had to be increased to 120,000 lb. per sq. in. (84.32 kg. per sq. mm.) to break the wire. Another wire was then stressed to 137,000 lb. per sq. in. (96.26 kg. per sq. mm.) at room temperature and immersed in liquid air while under stress, but the load had to be increased to 177,000 lb. per sq. in. (124.37 kg. per sq. mm.) to cause rupture. Another wire was stressed to 8000 lb. per sq. in. (5.61 kg. per sq. mm.) and immersed in liquid air under stress, and its breaking load was 73,500 lb. A final test was made in the usual manner, that is, without stressing at room temperature, and the tensile strength was 60,000 lb. per sq. in. (42.16 kg. per sq. mm.) or approximately what had been observed previously.

This wire is as brittle as any metal can be, so the explanation for the above phenomenon would not seem to be associated with permanent deformation. Besides, even a load of 8000 lb. per sq. in. applied at room temperature seemed to increase the breaking load over 10,000 lb. per sq. in. in liquid air. Also, the breaking load obtained on the equiaxed sample in this manner was just equal to the maximum breaking load observed on the strongest of the worked tungsten wires at  $-190^{\circ}\text{C}$ . I am at a loss to account for this phenomenon. More experimental information should be obtained to fix this irreversible relationship quantitatively.

*Reduction of Area of Tungsten.*—The tungsten wires all show 0.00 per cent. reduction of area at liquid-air temperatures. The reduction of area of the equiaxed sample is zero up to and including  $100^{\circ}\text{C}$ . It gradually increases above  $100^{\circ}\text{C}$ . with rising temperature and apparently reaches a maximum at about  $750^{\circ}\text{C}$ ., although the number of samples tested and the difference in structure noted microscopically did not warrant the extension of the curve above  $750^{\circ}\text{C}$ . The reduction of area of the worked wires increases as the temperature increases within the range tested.

## MECHANICAL PROPERTIES OF IRON WIRE

*Elongation of Iron Wire.*—Annealed iron wires with two different grain sizes were tested; Sample I-2 measured 810 grains per square millimeter and I-12, 2680 grains per square millimeter. I-2 was comparatively brittle at the temperature of liquid air and I-12 was ductile. The tests were not carried far enough to state with certainty that this difference in ductility at the low temperatures was entirely due to the

difference in grain size. In general, however, the smaller the grain size the greater the path for intercrystalline fracture and, hence, the lower the temperature at which intercrystalline fracture will take place. It is probable that, like tungsten, most metals will become brittle in the annealed condition at very low temperatures. Iron seems to reach this condition at about the temperature of liquid air. The fracture of some of the grains of I-2 was intercrystalline. This accords with the observations on tungsten. It is also evident that the grains of iron are deformable at liquid-air temperature, else I-12 could not have had 11.75 per cent. elongation in 2 in. at that temperature. If, therefore, iron is brittle at liquid-air temperatures, it is not because the grains are brittle but because the intercrystalline material, for some reason, probably internal stresses due to differences in expansion, gives way before a load sufficient to deform the crystalline material can be reached in tension. The manner in which the strong brittle amorphous phase protects the grain itself from deforming has been interestingly worked out by Humfrey.<sup>16</sup> He has observed that the grains deform in the interior more readily than at their boundaries. The lower the temperature the greater will be the reinforcement of the grains by the amorphous cement, and hence a greater load can be applied to a piece of metal taken as a whole before deforming the grains than would be represented by the resistance to deformation of an isolated grain at the same temperature. It therefore seems probable that the brittleness of iron observed by Hadfield at the temperature of liquid air was largely intercrystalline. At temperatures considerably below  $-190^{\circ}$  C. it might be expected that all annealed iron samples would be brittle and that the fracture would be intercrystalline. It might be of interest to note that, on cooling, molybdenum becomes brittle near room temperature and in its brittle region its fracture is intercrystalline.

A general confirmation of the proposition that a sharp increase in tensile strength accompanies brittleness produced by intercrystalline fracture is found in samples I-2 and I-12 having tensile strengths in liquid air of 109,500 and 105,500 lb. per sq. in. (76.65 and 73.85 kg. per sq. mm.) respectively. I-2 was the coarser grained and the fracture was partly intercrystalline. Of four samples tested, the stronger were the ones having least reduction of area and hence most nearly approaching complete intercrystalline fracture.

The elongation of annealed iron increases as the temperature increases to room temperature. At  $100^{\circ}$  C. its elongation reaches another minimum and gradually increases to a temperature of  $450^{\circ}$  C. This discontinuity in the elongation-temperature curve is also observed in the tensile strength and reduction of area curves. (See Fig. 62.)

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<sup>16</sup> J. C. W. Humfrey: Influence of Intercrystalline Cohesion upon the Mechanical Properties of Metals. Iron and Steel Institute, *Carnegie Scholarship Memoirs* (1913) 5, 86-99.

The elongation of annealed iron varies at all temperatures with the grain size; small grains favor high elongation. Fig. 62 shows the variation in elongation of annealed iron with variation in grain size and in the rate of loading. A decrease in elongation was not found between 800° C. and 900° C. corresponding to the brittle zone in Armco iron reported by Brooke and Hunting.<sup>17</sup> There seemed to be an increase in elongation in this range rather than a decrease.

Speller<sup>18</sup> also shows an increase in elongation in low-carbon steel at 800° C. The temperature region between 800° C. and 900° C. is the region of most rapid grain growth in iron between its recrystallization temperature and 1050° C. Its increased elongation may be due to a spontaneous annealing during test or to the fact that beta iron does not have the same elongation as alpha iron.

The elongation of the iron wires that have been worked below their annealing temperatures seem to show one common characteristic, namely, their elongation decreases above room temperature up to 100° C. Between 100° C. and 330° C., the elongation increases, then decreases again from 330° C. to 450° C. at which temperature an incipient annealing seems to cause a general increase in elongation.

The interpretation that I make of this phenomenon assumes the existence of an additional allotrope in iron.<sup>19</sup> It is probable that the change from one allotrope to the other is gradual with change in temperature because it occurs in a region that does not permit grain growth. It seems that the allotrope is in the process of transformation between room temperature and about 400° C. or 450° C. The transformation may not be complete even at room temperature. With this assumption, the elongation curves of worked iron below the annealing temperature obey the laws given above for copper and tungsten within the temperature range of any one allotrope. In the transformation range between the two allotropes the elongation will vary in an irregular manner. The elongation in the allotrope that exists between 400° C. and beta iron is greater than that of the allotrope stable at room temperature. This is shown by the fact that both samples I-2 and I-12 have greater elongation at 450° C. than at room temperature. It is also suggested by the fact that all the worked wires have higher elongation at a temperature between

<sup>17</sup> W. J. Brooke and F. F. Hunting: Note on the Microstructure of Commercially Pure Iron between Ar<sub>3</sub> and Ar<sub>2</sub>. *Journal, Iron and Steel Institute* (No. 2, 1917) **96**, 233-250.

<sup>18</sup> F. N. Speller: Discussion of paper by White and Wood, Recrystallization as a Factor in the Failure of Boiler Tubes. *Proceedings, American Society for Testing Materials* (1916) **16**, Pt. 2, 112.

<sup>19</sup> This has been suggested many times before but has never been strongly backed up nor accepted. The present evidence, it is thought, will be sufficient to establish the existence of the low-temperature allotrope beyond reasonable doubt.

330° C. and 450° C. than at room temperature. The elongation above the annealing temperature increases with the rate of loading but not to so marked a degree as in copper.

*Tensile Strength of Iron Wire.*—The general temperature-tenacity curve of annealed iron is shown in Fig. 61. It corresponds very well with the curve drawn from the combined data of Hadfield,<sup>20</sup> Huntington,<sup>21</sup> and Rosenhain and Humfrey;<sup>22</sup> it is also in good agreement with Speller's<sup>23</sup> curve.

It will be considered that the increase in tensile strength between 0° and 200° C. is caused by allotropy. The reasons for this assumption are as follows: The tenacity will depend on the path of rupture along the amorphous slip planes generated during the tensile test and on the ability of the iron to break by degrees. The tenacity of the amorphous phase must decrease with increase in temperature and therefore the path of rupture along the amorphous planes must be much greater at 200° C. in iron than it is at room temperature. This greater path of rupture must be caused by a difference in the deformational properties of the crystalline phase, that is, the slip planes must form at different angles. The resistance to deformation of the crystalline phase should also be greater to produce the greater path of rupture along the slip planes. This condition necessitates a different arrangement of the atoms within the grains of iron at 200° C. from that at room temperature. A different arrangement of the atoms can mean nothing short of allotropy. Of course we do not expect the grain size to change at this allotropic point because it occurs in a region in which grain growth is not possible in iron. The change must therefore be brought about by a change in the positions of the atoms within the grain itself. The amorphous sheath around any grain will remain intact during the transformation and the transformed grain on cooling will have the same boundaries as the initial grain. The grains we observe at room temperature are pseudomorphs. The maximum strength, which occurs between 200° C. and 300° C., should occur when the atoms are partly in the arrangement of one crystal form and partly in the other. This is a condition that should produce both a long path of rupture along the amorphous planes and greater uniformity of time of rupture of the various parts.

This fact is demonstrated nicely by the tenacity of the iron wires drawn below the annealing temperature. The wire reduced 96 per cent. by

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<sup>20</sup> *Op. cit.*

<sup>21</sup> A. K. Huntington: Effect of Temperature on the Tensile Tests of Copper and Its Alloys. *Journal, Institute of Metals* (No. 2, 1912) 8, 126–148.

<sup>22</sup> Walter Rosenhain and J. C. W. Humfrey: Tenacity, Deformation and Fracture of Soft Steel at High Temperatures. *Journal, Iron and Steel Institute* (No. 1, 1913) 219.

<sup>23</sup> *Op. cit.*

drawing at room temperature (I-1) is not as strong at any temperature above as it is at room temperature. The discontinuity in the tenacity curve, however, is evident. The amount of deformation on this wire has been so great at room temperature that a considerable amount of amorphous material has been generated within the deformed grains. On heating this wire to 200° C., the amorphous phase becomes weaker than at room temperature but the crystalline phase increases its resistance to deformation due to the change into the new allotrope. The tensile strength at 200° C. will therefore be a weighted average between the decrease in strength due to the amorphous phase and the increase due to the crystalline phase. Since the amorphous phase controls as regards rupture this wire does not become stronger with increase in temperature. Due to the added resistance to deformation in the crystalline phase, however, and to the fact that the small crystalline fragments within the deformed grains must deform to a certain extent before the wire can break at 200° C., the tensile strength has not decreased as much as would be indicated by the decrease in strength in the amorphous phase alone.

On the other hand, the wires that were drawn at 275° C. were deformed in a region in which deformation caused the generation of amorphous material along irregular paths, that is, the path of rupture along the amorphous planes would be large. Consequently sample I-10, which was reduced by drawing at 275° C. only 32 per cent. in area, was not only stronger than I-1 at the higher temperatures but was also stronger at room temperature. During the deformation at 275° C., a long path of rupture along the amorphous planes was produced and on cooling to room temperature this amorphous phase increased in cohesion but the crystalline fragments decreased in cohesion. The amorphous phase being the controlling one makes this wire stronger at room temperature than I-1.

The marked increase in strength of iron on cooling from 450° C. to 330° indicates either a rapid increase in the path of rupture along amorphous planes or a more simultaneous rupture. Either of these might be the result of a more or less chaotic arrangement of the atoms, that is, atoms partly in the arrangement of one allotrope and partly in the other. All of these facts strongly suggest that the process of deformation in iron between room temperature and 450° C. produces a long path of potential rupture through the amorphous metal.

There seems to be no discontinuity in the specific heat or coefficient of expansion of iron in the region of this allotrope. There is, however, a discontinuity in the electrical resistivity.<sup>24</sup> It is probable that there

<sup>24</sup> A. R. Meyer: Über die Änderung des Elektrischen Widerstandes reinen Eisens mit der Temperatur in dem Bereiche, 0° bis 1000° C. *Berichte Deutschen Physikalische Gesellschaft* (1911) 9, 680.

may be discontinuities in the other properties but when the difference is distributed through a temperature interval of 400° C. the discontinuity may be masked.

The tensile-strength curves of iron below the annealing temperature furnish a striking verification of the hypothesis that the mechanical properties of a metal can be accounted for by the variation of the mechanical properties of the two phases and the quantity and arrangement of each within the metal. No matter how much work is put on iron at room temperature the metal shows a discontinuity in tensile strength above room temperature, but its discontinuity is not as marked as in annealed iron; this is in accordance with the proposition that the properties of the amorphous phase are not affected by allotropy, that the amorphous phase is increased in quantity by cold working, and that a large part of the metal within a deformed grain is crystalline.

A gradual decrease in tenacity was observed in passing from the alpha to the beta condition, but a marked increase in tenacity was observed in passing from beta to gamma iron. This increase must again be interpreted as a condition that makes a longer path of rupture through the amorphous phase in the gamma iron than in the beta iron coupled with a higher resistance to deformation of the crystalline phase. Gamma iron was found to be about 25 per cent. stronger at 1000° C. than beta iron at 835° C. It is obvious that the crystalline phase of gamma iron has a higher resistance to deformation than the crystalline phase of beta iron.

Grain growth seems to be determined, to a large extent, by the hardness of the material. Grain growth will take place more easily in beta iron just below the transformation range than in gamma iron just above the transformation range. This grain refinement can only be responsible, however, for a small part of the increased tenacity in the gamma iron; the major portion must be due to the generation of a long potential path of rupture within the gamma iron by deformation. This is analogous to the rapid hardening of manganese steel by cold deformation. Manganese steel has the same crystal structure as gamma iron. If a small amount of deformation hardens manganese steel markedly, it must at the same time generate a relatively large amount of amorphous material for a given amount of deformation.

The rate of loading affects the tensile strength of iron in a manner similar to that described for copper. No attempt was made to determine the effect of variation in the loading rate at the lower temperatures. Since making these tests, however, I note that Dalby<sup>25</sup> has reported the difference in properties between iron tested with rapid and slow loading

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<sup>25</sup> W. E. Dalby: Researches Made Possible by the Autographic Load-extension Optical Indicator. *Journal, Institute of Metals* (No. 2, 1917) **18**, 5-43.

at room temperature. He finds very little difference in the tenacity but finds that the elongation in 5 in. increases from 24 per cent., with slow loading, to 29 per cent., with rapid loading.

The reason allotropy is supposed to be caused by changes in the positions of the atoms rather than by changes within the atoms is that the strictly atomic properties do not seem to be affected by temperature in a manner at all comparable with the changes of the interatomic properties. Radium, for example, has the same rate of emanation in all physical and chemical states. Since differences in temperature represent differences in energy of motion of the atoms, the cohesion properties would be affected greatly by change in temperature, whereas the atomic properties might be affected little or not at all. We might compare radium atoms to incandescent lamps. If we suppose that the light given off by the incandescent lamps corresponds to the emanation from the radium atoms, a group of these lamps at rest with the electric current turned on will correspond to a group of radium atoms at absolute zero. If we set the lamps in motion, their kinetic energy will correspond to the kinetic energy of the radium atoms caused by an increase in temperature. The quantity of light given off by the lamps in motion will be the same as the quantity given off at rest and consequently the kinetic energy of the lamp taken as a whole will not affect the rate of light emanation. If the emanation depended on the kinetic energy of the atoms, it would be an interatomic property and would vary with the temperature. All of this indicates that the radium emanation is an atomic property and hence not affected by temperature, at least within the ranges so far tested. Since allotropy is governed by temperature, it is assumed that it is an interatomic property rather than atomic.

Of course the primary cause for allotropy must be attributed to certain properties of the atoms, but this should be due to interatomic forces that are probably directional and give the atoms the ability to form in the regular arrangement of the crystalline phase. Since the interatomic forces permit different positions of the atoms relative to one another as their kinetic energy changes, it is logical to assume that certain positions coupled with given atomic forces would produce an entire change in the equilibrium and hence a new system of arrangement of the atoms would produce what we call allotropy. If allotropy depends on a systematic arrangement of the atoms, the amorphous phase cannot be allotropic because the atoms are arranged in a random manner.

The rate of transformation from one allotrope to another should depend on the cohesion of the metal at the temperature at which the change should take place. If the temperature is high and the cohesion low, the change will be effected easily; but if the temperature is low and the cohesion high, the change will take place reluctantly. It seems that the low-temperature allotrope in iron is in the process of change from

a temperature around 400° C. to room temperature, whereas the high-temperature allotropes change completely from one to another either at a given temperature or within a very small temperature range.

In the transformation of the low-temperature allotrope, there should be two opposing forces; one, the force tending to produce the change that will be greater the lower the temperature; the other, the force tending to resist the change, that is, the force of cohesion that will be greater the lower the temperature. If the latter force increases at a faster rate than the former and is greater than the former, the transformation will not take place. However, when iron is cooling from 400° C. to room temperature, its cohesion increases until a temperature of about 250° C. is reached, below which it decreases until room temperature is reached. But this decreased cohesion is caused by the allotropic change, so we may conclude that the force tending to produce the change in pure iron is greater than the force resisting it.

When we increase the cohesion of iron by cold working or by the introduction of alloying elements, we increase the force resisting allotropic transformation and may prevent it. This is a plausible explanation for the change in elastic limit and tensile strength of hard-drawn iron and steel by heating to a temperature in the neighborhood of 300° C. If the iron or steel has been drawn cold, the amorphous phase will have been greatly increased in quantity and hence the cohesion will have been increased. Each fragment of crystalline material that is then in the form of a softer allotrope will, when the metal is heated to 300° C., change over, at least partly, into the stronger allotrope. The resistance to change from a soft allotrope to a hard one should be less than the resistance to change from a hard allotrope to a soft one, for the reasons given. The change from the soft to the hard allotrope will have been in process while the metal has been heating and while soaking at the highest temperature reached; this will be the temperature of the least cohesion and hence it will offer the least resistance to the change in position of the atoms. The cohesion will actually increase at constant temperature as the allotropic change progresses. As soon as cooling begins, the cohesion is increased still further, hence the resistance to the reversed allotropic change increases. On reaching room temperature, the crystalline fragments may not have changed back to the soft allotrope, which would be the reason for the increased elastic limit and tensile strength.

Even some of our alloy steels that have been quenched from above their critical ranges and tempered at a temperature under 600° C. have probably retained the iron in the form of the hard low-temperature allotrope. The annealed alloy steels of the same composition, however, will be soft enough to have permitted the change into the softer allotrope. The following tests by W. E. Ruder on a nickel-chromium steel substantiates the above. This steel in the annealed condition had a tensile



strength of 101,500 lb. per sq. in. (71.05 kg. per sq. mm.). When the annealed sample was reduced cold 10 per cent. in area, its tensile strength was 114,200 lb. per sq. in. (79.94 kg. per sq. mm.). When the same steel was reduced the same amount at 300° C., its tensile strength at room temperature was 137,600 lb. per sq. in. (96.32 kg. per sq. mm.). This shows that the annealed sample contained the free iron at room temperature largely in the form of the soft allotrope and that at 300° C. the hard allotrope had formed and the deformation was produced in it. The cohesion may have been increased at 300° C. enough to have prevented some of the change from the hard allotrope to the soft on cooling to room temperature. However, since the cohesion of the hard allotrope is actually greater at 300° than that of the soft allotrope at room temperature, the difference in quantity and arrangement of the amorphous phase produced by the deformation must have played a large part in this increased cohesion.

On the other hand, when the heat-treated alloy steel that has been tempered under 600° C. is deformed at room temperature, it is found that the increased cohesion is nearly the same as if the deformation had been effected at 300° C. This strongly suggests that the iron is in the same form at room temperature in the heat-treated steel as at 300° C. and that at both temperatures the iron is in the form of the harder allotrope.

*Reduction of Area of Iron.*—The reduction of area of annealed iron increases as the temperature increases from  $-190^{\circ}$  to  $100^{\circ}$  C. It shows a discontinuity between  $100^{\circ}$  and  $450^{\circ}$  C. but the temperature of minimum reduction of area, as shown in Fig. 63, does not correspond to that of minimum elongation, as shown in Fig. 62. The reduction of area increases as the grain size decreases at all temperatures. Above the annealing temperature, the reduction of area also increases with the rate of loading. There is a general decrease in reduction of area above  $575^{\circ}$  C.

All of the Armco iron wires drawn below the annealing temperature show discontinuities in the reduction of area curves between room temperature and  $450^{\circ}$  C. It is thought that these discontinuities are caused by allotropy, as discussed in connection with the elongation and tensile strength.

#### EFFECT OF DIAMETER OF WIRE ON MECHANICAL PROPERTIES

The smallest wire tested was sample I-12, which had a diameter of 0.023 in. (0.5842 mm.); the largest wire was T-4 with a diameter of 0.028 in. (0.7112 mm.). The rest of the samples were 0.025 in. (0.635 mm.) in diameter. It is logical to suppose that the structure of a small wire is more uniform than that of a piece of the same material with a larger cross-section. The deformation in cold working can be extended to the

axis in a small wire more easily than in a large wire. On the other hand, the non-metallic inclusions, like slag in a metal, do not extend during cold drawing; the metal is drawn around them. These become relatively larger, therefore, as the wire becomes smaller, and hence their influence on the mechanical properties is proportionately greater in a small cold-drawn wire than in a larger hot-rolled or hot-forged piece. In small annealed wires the mechanical properties are not the same as in large wires with the same grain size. Table 26 illustrates this point.

TABLE 26.—*Effect of Diameter and Grain Size on Mechanical Properties of Annealed Armco Iron*

Diameter, In.	Grains per Sq. Mm.	Tensile Strength, Lb. per Sq. In.	Elongation in 2 In., Per Cent	Reduction of Area, Per Cent.	Number of Grains Across Diam. of Wire
0 204	400	41,000	30 0	76 0	100
0.025	810	48,550	12 5	76 0	18
0 023	2680	48,200	23 7	78.5	30

The number of grains exposed on the whole cross-section of the wire is more of a criterion as regards elongation than the actual grain size. It is obvious that with any given grain size we could have a wire so small that its whole cross-section would be occupied by one grain. No matter how small this wire nor how small the grains composing it, its properties would never approach those of a larger wire composed of grains of the same size. Glass, when broken in tension at a red heat with rapid loading, may show several hundred per cent. elongation. This may be considered analogous to a metal with as many grains on a cross-section as there are atoms on the cross-section of the piece of glass. The analogy is not perfect because the atoms do not permanently deform.

The mechanical properties of an annealed metal will depend on the actual grain size and the number of grains on a cross-section. With grains of a given size, the larger the cross-section of the piece of metal the more will the random orientations tend to counterbalance the directional properties of the grains.

A metal rod with a diameter equal to that of the earth might be composed of grains as large as houses and still its elongation when broken in tension would be greater than that of a wire 0.001 in. (0.0254 mm.) diameter composed of grains about 0.001 in. diameter. It is probable that the variation in diameter of a wire will cease to be important as regards variation in elongation with a given grain size when the number of grains on a cross-section is sufficient to nullify the general directional properties. The elongation in a given length will always be influenced by the diameter of the wire or rod on account of the tendency of most metals to "neck" at the point of fracture. The greater the necking and the larger

the piece, the greater will this factor influence the measured elongation in a given length.

The number of grains on a cross-section of a wire or rod also influences the reduction of area. As previously mentioned, cold-worked copper often shows a greater reduction of area than annealed copper. The reason for this is probably the great number of deformed grains of copper on a cross-section of the test piece as compared to the fewer grains on the annealed piece. If two pieces of copper have the same number of grains on a cross-section and one piece has been cold worked and the other annealed, the latter will have the greater reduction of area.

A small cross-section seems always to favor high reduction of area. This is probably due to the fact that the surface portions have so much farther to travel when a large piece "necks."

Certain factors should be directly dependent on the grain size irrespective of the cross-section of the piece of metal: These are the degree in which the intercrystalline amorphous film (assuming a given thickness) increases the resistance to deformation of the interior crystalline portion and the internal deformation of a grain of given size, orientation, and direction of load.

#### GENERAL CONSIDERATIONS ON PROPERTIES OF METALS AT VERY LOW TEMPERATURES

Since the high-melting-point metals, like tungsten and molybdenum, become brittle on cooling to room temperature and iron approaches brittleness at liquid-air temperature, it might be concluded that all metals will be brittle at some very low temperature. F. A. and C. L. Lindemann<sup>26</sup> have made tensile tests (for tenacity only) in liquid air ( $-192^{\circ}\text{C.}$ ) and liquid hydrogen ( $-252.6^{\circ}\text{C.}$ ) on very small wires (0.05 mm. diam.) of aluminum, iron, nickel, copper, silver, gold, and platinum and on a wire 0.11 mm. diameter of lead. The authors make the sweeping statement that "the wires became very brittle at low temperature and showed a marked tendency to break so that these measurements are only accurate within about 10 per cent. Zinc and bismuth were so brittle that they could not be measured at all." Unfortunately, the wires, with the exception of lead, must have been cold worked and some of them in addition were alloyed with strengthening metals. This is strongly indicated from the results at room temperature. We know from Hadfield's results and those reported in this paper that all these metals are not brittle in liquid air. Will they become brittle in liquid hydrogen or at still lower temperatures? Must all metals become brittle at absolute zero?

Probably most ductile metals will become brittle as absolute zero is

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<sup>26</sup> Note on the Tensile Strength of Materials at Low Temperatures. "Festschrift, W. Nernst," 264-65. Halle a.d.S., 1912, W. Knapp.

approached for the same reasons that tungsten becomes brittle at room temperature. There is no physical reason apparent, however, why a metal could not be deformed near absolute zero. The criterion would be its ability to generate amorphous metal at the slip planes.

Furthermore, a crystal that assumes cleavage brittleness at a certain low temperature may be deformed under high pressure. A crystal of brittle calcite, for example, can be enclosed in a steel tube and can then be deformed when the tube is rolled. The statement can be made, therefore, as a prediction subject to further verification, that if no metallic crystals are deformable near absolute zero under ordinary pressure they can be deformed at that temperature under sufficiently high pressure. Also, the generality should hold that all metals that assume intercrystalline brittleness at low temperatures will retain ductility at lower temperatures in the fibrous (cold-worked) than in the annealed state.

#### GENERAL CONSIDERATIONS ON PHYSICAL PROPERTIES OF COMPLEX ALLOYS

A careful analysis of the properties of any alloy composed of more than one metallographic constituent can be made by the general method described for the single constituent metals. The properties of each component and each phase must be ascertained and the arrangement and quantity of each phase will then serve as a guide to interpret the properties of any mixture taken as a whole.

Let us consider the mechanical properties of high-carbon annealed steel in which a network of cementite surrounds pearlite. The two main entities are cementite ( $\text{Fe}_3\text{C}$ ) and ferrite (pure iron). The cementite is partly in the network and partly in thin sheets alternating with somewhat thicker sheets of ferrite to form pearlite; all of the ferrite is in the pearlite. Cementite is hard and undeformable, while the ferrite is soft and ductile. If there are grains of cementite, we would expect amorphous iron carbide between them. If any grains of ferrite are present with different orientations abutting one another, there will be amorphous iron between them. What exists at the junction between ferrite and cementite? I believe an amorphous solution of iron carbide in iron. This solution may gradually enrich in iron near the ferrite and in iron carbide near the cementite. There are therefore two crystalline materials and three amorphous materials present, no two of which have the same properties at any given temperature nor obey the same laws of change in properties with change in temperature. There are also many possible variations in the quantities and arrangements of these constituents. It is no wonder that high-carbon steel is a treacherous alloy and is noted for its great variety of mechanical properties under different treatment conditions.

The weakness of high-carbon steel having a cementite network structure is no doubt due to internal stresses caused partly by the different coefficients of expansion of cementite and pearlite. Fracture takes place at the cementite network. Cementite is practically undeformable. Ferrite is very ductile. Pearlite made up of alternate sheets of cementite (13.5 per cent.) and ferrite (86.5 per cent.) is somewhat deformable because the path of rupture along the cementite-ferrite junctions is so great that the plates of cementite break during the early stages of rupture. This forces the ferrite to take the load, and it will not rupture without deformation. The net result after complete rupture is a total deformation greater than that of cementite but less than that of ferrite. When the cementite of the pearlite is changed from sheets into globules by heat treatment, the pearlite becomes very ductile. The ferrite deforms and leaves the cementite globules intact much the same as if they were slag.

The modulus of elasticity of each constituent will play a rôle in the interpretation of the mechanical properties of alloys. Young's modulus itself may not be so important as the actual amount of stretch possible before permanent deformation begins.

The chief value of the proper interpretations of the properties of metals and alloys is the assistance rendered in making predictions as to their properties under various conditions. If the interpretations are correct, the predictions will in the main be correct. Space does not permit the further discussion of this important aspect of the subject but it is of interest to note that some predictions from the general data outlined in this paper have been made and the results have been surprisingly accurate.

The method of attack, which these ideas suggest, on the phenomena encountered in solid solutions will be pointed out. A solid solution consists of two phases, crystalline solid solution and amorphous solid solution. Why is a solid solution harder than a pure metal? Certainly the amorphous solid solution is harder than the pure amorphous metal; the crystalline phase probably is also. But the melting point of a solid solution is usually lower than that of the higher-melting-point component, so within a certain high temperature range the cohesion of the amorphous solution is less than that of one of the pure amorphous metals. Conditions are reversed at low temperatures. Therefore the rate of increase in cohesion with decrease in temperature is greater in a solid-solution amorphous phase than in a pure-metal amorphous phase. If physicists can explain this fact we need look little further for the explanation of the cohesion properties of solid solutions. But it is also probable that the solid-solution crystalline phase shows a similar increase in cohesion above that of the pure-metal crystalline phase on cooling from a high temperature. Both of these statements are subject to experimental verification. As soon as the physicists can explain the greater rate of change

of cohesion with change in temperature in solid solutions they will have explained what is now a mystery, namely, the greater cohesion. This explanation should also account for the observed changes in electric and heat conductivity.

The following suggestions relate to pure components and solid solutions: The amorphous phase contains more heat energy at the melting point than the crystalline phase because the latter has given up its heat of crystallization. Assuming that both phases contain no heat at absolute zero, the amorphous phase must lose heat energy on cooling at a faster rate than the crystalline. But the rate of losing heat energy controls, to a certain extent, the rate of change of cohesion. This, coupled with the random arrangement of the atoms, probably accounts for the greater rate of change of cohesion in the amorphous phase.

Other apparent factors influencing the cohesion of a solid solution are the different atomic forces and the different size and, possibly, shapes of the atoms. These factors should influence both the amorphous and crystalline phases. We would expect greater cohesion in a crystalline material made up of atoms of different size than in one composed of atoms of the same size. The same is true of the amorphous phase.

#### SUMMARY

A MORE complete interpretation of the amorphous theory in metals is given than has heretofore been offered. It is believed that Le Chatelier, Tammann, and Heyn, at present the ablest opponents of the amorphous theory, will not be able longer to continue their beliefs that surface tension alone can account for all the observed variations in the properties of single constituent metals.

It is strongly advocated that the properties of any solid substance be studied from the standpoint of the number of constituents present, the quantity and arrangement of each constituent, and the properties of each constituent. Each deformable crystalline constituent by plastic deformation will produce amorphous material, thus changing the properties.

Considerable attention is given to the variations in properties of the amorphous and crystalline phases of any substance. These are summarized as follows:

1. The cohesion of the amorphous phase of a substance is substantially zero at the melting point while the cohesion of the crystalline phase is considerable at the melting point. With decrease in temperature, the cohesion of the amorphous phase increases at a faster rate than that of the crystalline phase; this differential rate of change of cohesion holds between absolute zero and the melting point. At a certain temperature for each substance, the cohesion-temperature curves of the amorphous and crystalline phases intersect. In most ductile metals, this intersection

occurs at about 0.35 to 0.45 of the absolute melting point. This point of intersection has been called the "equi-cohesive" temperature; below it the amorphous phase is not only more cohesive than the crystalline but it increases in cohesion with decrease in temperature at a faster rate than the crystalline phase. Above the equi-cohesive temperature, the amorphous phase is not only weaker than the crystalline phase but its cohesion decreases with increase in temperature at a faster rate. The equi-cohesive temperature corresponds closely to the lowest recrystallization temperature of a metal after severe cold work. Recrystallization seems to be caused by grain growth and grain growth does not seem to take place until a temperature is reached at which the amorphous phase is softer than the crystalline. These facts, coupled with a knowledge of the arrangements and relative quantities of the amorphous and crystalline phase, enable one to account for the observed mechanical properties of a metal.

2. The coefficient of expansion of the amorphous and crystalline phases of a substance, in general, should vary inversely as their cohesion. The rate of change of expansion with change in temperature should be great in the amorphous phase and small in the crystalline phase. There should be a temperature of equal expansion but this may not correspond to the temperature of equal cohesion. The same analogy may be considered for the modulus of elasticity and other related properties, such as compressibility.

The difference in coefficient of expansion between the amorphous and crystalline phases may cause intercrystalline brittleness in a metal at low temperatures. The temperature at which a metal becomes brittle on cooling can be made lower by changing the arrangement of the amorphous phase into a less commanding position. For example, equiaxed tungsten becomes brittle between 100° and 200° C. on cooling. By changing the equiaxed to a fibrous structure, the metal retains useful ductility at and somewhat below room temperature.

3. If a non-allotropic metal is worked below its recrystallization temperature, its elongation and tenacity increase below the temperature at which it was deformed and decrease above it up to the recrystallization temperature.

4. The rate of deformation of the amorphous phase with a given absolute cohesion is much less than that of the crystalline phase at a temperature of equal cohesion. At the equi-cohesive temperature, for example, the amorphous phase deforms slowly under a given load while the crystalline phase deforms quickly. The measured equi-cohesive temperature will be higher, therefore, the shorter the time of test. This causes the strength, elongation, and reduction of area of metals tested above their recrystallization temperatures to increase. The amorphous phase may remain within its elastic limit with a short duration of a

given load and deform with long duration; the permanent bending of glass rods and tubes after resting months or years in bent positions would be due to this factor. The same pieces of glass could be bent at room temperature and would come back to their original positions if they were held in their bent positions a short time only. The lag in expansion and contraction of such hard amorphous materials as fused silica is no doubt caused by this same phenomenon.

5. This idea that the amorphous phase deforms at a slower rate than the crystalline is not consistent with the idea that the amorphous phase itself passes through a mobile state during deformation. It is more probable that the mobile state occurs while the amorphous phase is in the process of formation at the slip planes; the mobile state is the one permitting a slip and this generates amorphous material. In overcoming the momentum of the slip, the atoms of the amorphous phase are probably left in a state of strain immediately after deformation. The slow adjustment of the strained amorphous metal should account for the observed changes in properties with age, in such metals as iron. The same reasoning holds for the amorphous phase at grain boundaries. Adjustment of atoms and removal of strain take place after an annealed metal is cooled; in hard metals this change will be slow and in soft metals rapid.

6. The mechanical properties of iron can be explained best by assuming that an allotropic change occurs between room temperature and 450° C. All the tensile properties of both annealed and cold-worked iron show discontinuities in this temperature range. Annealed iron is 20 to 25 per cent. stronger between 200° and 300° C. than at room temperature. This increase in strength can be imparted to the metal at room temperature by working in the region of the stronger allotrope. It is assumed that allotropy is associated with the crystalline phase only, that is, it is caused by a change in the arrangement of the atoms. If allotropy proves to be due to a change within the atom, these views can be easily adjusted to the new idea.

7. The mechanism of rupture is discussed. If a crystal breaks without deformation, it is probable that no amorphous material is generated at the planes of fracture. The ability of a crystal to be deformed depends absolutely on its ability to generate amorphous metal at points of incipient rupture. Rupture takes place by degrees, that is, all parts of the fracture do not separate at the same time; ductility favors rupture by degrees. The reduction of area at the point of fracture is a result of rupture by degrees. Rupture by degrees also favors a low breaking load but at the same time it tends to make a long path of rupture. The balance of these two tendencies will control the breaking load and reduction of area and hence the elongation. The amorphous phase has the ability to flow like glass at relatively high temperatures but, like glass, it is rigid and brittle at low temperature.



8. The results of several hundred tensile tests on copper, tungsten and iron at temperatures between  $-190^{\circ}$  and  $900^{\circ}$  to  $1000^{\circ}$  C. are discussed. The results are summarized in the graphs shown in Figs. 43 to 73 inclusive.

9. A brief note is given dealing with the interpretation of the properties of complex alloys. A suggestion as to the method of attack on the solid-solution mystery is given.

*Acknowledgments.*—I wish to express my indebtedness to Mr. W. P. Sykes, who assisted with the tensile tests recorded. I wish to thank Prof. C. L. Norton and Mr. G. B. Wilkes for valuable assistance in connection with the liquid air tests. Finally, I wish to thank Mr. B. L. Benbow and his staff at the Cleveland Wire Division of the General Electric Co. for valuable assistance in connection with the production of the wire samples and the design and construction of testing apparatus.

## DISCUSSION

C. H. MATHEWSON, New Haven, Conn. (written discussion\*).—In a recent discussion of Dr. Jeffries' paper on tungsten,<sup>1</sup> J. C. W. Humfrey, after taking exception to certain of the author's ideas relative to the cohesion of *A* and *C* metal, expresses the opinion that future research is likely to develop a method of preparing tungsten possessing "mechanical properties in line with those of other metals." The present paper by Dr. Jeffries begins with a determination of comparative properties of tungsten, copper, and iron, in which much dissimilarity is apparent, and ends with a coherent explanation as to why much of this dissimilarity is natural and inevitable, thereby supplying theoretical means for visualizing the changing properties of metals in general, once certain fundamental facts are known.

The author's reasoning is primarily from the standpoint of the amorphous theory and his principal achievement is the development of an idea of differential cohesion according to conditions of temperature, etc., between *A* and *C* metal (which was first plainly suggested by Rosenhain and Ewen, in their papers on Intercrystalline Cohesion of Metals) along with associated ideas, so as to give us a really useful working theory.

I have come rather reluctantly to believe in the amorphous theory, or some theory admitting a modification of the ordinary crystalline phase so as to permit differential properties within the conglomerate, mainly because in no other way does it seem possible to account for observed changes of properties with temperature, etc. The very best illustration of the competency of the amorphous theory in this respect is seen in this and other recent papers by the author. All fractures through normal or healthy metal that have been preceded by deformation are supposed,

\* Received Feb. 17, 1919.

<sup>1</sup> This volume.

by Dr. Jeffries, to take place either through the amorphous phase or at certain boundaries between amorphous and crystalline material; his reference to fractures, both members of which are similar in appearance and do not reveal a preëxisting coarse-grained structure because of a coating of amorphous metal, indicates his preference for the former interpretation.

It is difficult to imagine any structure made up of crystalline grains surrounded by a truly amorphous cementing material—that is, any structure in which there is an abrupt transition from a crystalline orientation of particles to a truly amorphous orientation—which will not show preponderating weakness at the junction between phases. On the other hand, it is easy to imagine a structure in which there is material between neighboring grains wholly belonging neither to one nor the other, but passing from one orientation through a more or less neutral position or subcrystalline condition to the other orientation, and such a structure might well reach its greatest strength in the boundary region.

Some detail of this sort appears to be necessary because the majority of Dr. Jeffries' conclusions are based on a process of weighting averages from a specific assignment of properties to an amorphous component and a crystalline component. Ordinarily, allowance would need to be made for the cohesion or attraction between these phases, but on the above assumption of continuity this would not seem necessary, as every property would pass from one extreme to the other without discontinuity.

Dr. Jeffries' cites cold coarse-grained zinc as a brittle metal that, at the fracture, shows grains reflecting light directionally owing to an absence of amorphous metal on the surface of fracture. Cold zinc is an admirable example of something that must be placed in a class intermediate between "easily deformable" and "brittle." It can be worked cold, but with greater expenditure of energy than when heated and with greater likelihood of complicated fracture before the grain becomes materially refined. If all the elements of fracture through different crystals are brought into one plane they cannot be distinguished from one another either by direct observation with any variety of illumination or under any form of microscopic examination. Either the fractures are all through one set of cleavage planes or the directional irregularities of the surface are so minute that the reflection of light takes place as it would from an ideally plane surface. I am thus of the opinion that no conclusion can be drawn as to the presence or absence of an amorphous film by simple examination of fractured surfaces. Directional characteristics are revealed only after etching, straining to produce slip bands, or like methods. Even some forms of etching fail absolutely to develop the minute etching pits, which collectively furnish the evidences of orientation but leave surfaces that appear smooth under the highest magnifications. It is quite easy to develop a coarse-grained structure in a piece of rather pure

thin zinc sheet, originally of small equiaxed grain, by moderate local strain followed by annealing. If, after annealing, the piece is dipped momentarily in concentrated nitric acid, a surface effect similar to that obtainable by fine polishing is secured. It is inconceivable that any amorphous coating can remain on the surface as the dip can be repeated at will with the removal of any desired amount of metal from the surface and the effect remains the same.

It would appear that the tin fracture which Dr. Jeffries contrasts with the zinc fracture does not reveal the original coarse-grained structure because sufficient deformation has taken place during the tensile test to produce small grain fragments and complicate the path of rupture. The outlines of the original grains may be visible on a polished and etched section near the break because the fragments from one grain, although differently oriented, conform somewhat to the original orientation within the grain. I believe, with Dr. Jeffries, that the fracture is through amorphous or subcrystalline material, but see no way of demonstrating this visually.

There is little satisfaction in the conventional idea that the early process of rectilinear slip carries with it a generation of amorphous material. When an annealed metal begins to accommodate itself to an applied stress, movements either of simple translation or of rotation into twinning position occur and can be observed up to a certain point, as is well known. A coarse-grained and etched piece of zinc strip, for example, when stressed on the microscope stage shows instantaneous eruptions across the surface of a grain. Bands of variable, but usually quite appreciable, width leap across or up through the grain and lie inclined to the original horizontal plane of the grain. Unquestionably, a movement in accord with the general symmetrical relationships within the grain has occurred: probably a movement into twinning position. It seems to me that any unsymmetrical disturbance of the molecules must have been confined to the grain boundaries where several systems meet and conflict. Should there not be an increase in the amount of amorphous material at the boundaries with every movement of this sort?

The movements of simple translation are also instantaneous and give the impression that blocks of material have jumped from one position of equilibrium into another. I would also expect these movements to effect some sort of dislocation at the grain boundaries rather than along the planes of slip. As the stress builds up, these movements within the grain may well be expected to lose their simple character; perhaps the thickened or strengthened boundaries resist load from within and cause a kind of crumpling effect, which brings about a slight change of orientation from block to block within the grain. This is the beginning of a true fragmentation, which increases with the degree of deformation and ultimately, after very severe deformation, produces fragments that

in the case of brass, must measure more than 1,000,000 to the square millimeter because the first visible recrystallized units are at least as small as this.

I cannot see where our ordinary micrographic observations conflict with the above views. The first stages of fragmentation are not visible because the changes in orientation within a grain are slight and the appearance of etching pits will not suffice to bring about a distinction between fragments of nearly the same orientation.<sup>2</sup> It is my experience that, after severe deformation, the etching pits are similar in neighboring grains; they are no longer true etching pits and give no idea of orientation, as individual units of definite orientation are too small for observation.<sup>3</sup> The original grain boundaries may be even more prominent than before deformation because they are likely to etch selectively as the principal seat of the most electropositive (amorphous?) material in the conglomerate.

As near as I can judge, these views differ from Dr. Jeffries' ideas only in one essential respect: he believes that, during the deformation of annealed material below the equicohesive temperature, material within a given grain begins to be reinforced against further deformation by the development of amorphous metal along the slip-planes, immediately after the elastic limit has been exceeded; whereas I believe that the first reinforcement occurs at the grain boundaries and that no reinforcement begins within the grain until the grain itself is broken down into fragments of varying orientation. That this latter effect begins very soon is evident from the small amount of deformation necessary to bring about a visible refining of the grain on subsequent anneal or a visible coarsening if conditions are such as to induce selective growth; viz., the elastic limit cannot be raised greatly by such deformation as merely brings about movements of simple translation, and this form of deformation can hardly play any prominent part in reinforcing the grain-substance.

Dr. Jeffries states that the principal difference between annealed and hard-drawn metal (copper) is in the quantity and the arrangement of the amorphous phase. Regarding the amorphous phase as something more of a transitional product, a little more crystalline and a little less amorphous, this suits my own conception of the difference between worked and annealed metal. However, my idea of the arrangement of the amorphous or sub-crystalline phase in worked metal confers upon the latter characteristics that would cause it to resemble what might be called abnormally fine-grained annealed metal; that is, equiaxed metal free from any possible strain complication and finer grained than can be obtained in any ordinary way.

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<sup>2</sup> See Jeffries' remarks on this same subject in *Trans.* (1916) 56.

<sup>3</sup> Compare also J. Czochralski: *Internationale Zeitschrift für Metallographie* (1914) 6, 294.

I call to mind in this connection Bennett's<sup>4</sup> observations that hard-drawn copper can be deposited on a rotating cathode and likewise annealed copper by proper variation of current density, temperature, and speed of rotation. The tensile strength varied between 30,000 to 68,000 lb. per sq. in.; and although no photomicrographs were included, corresponding variations in grain size were especially noted. Hardness, slight ductility, and very fine grain were associated with high tensile strength; softness, pronounced ductility, and coarser grain, with low tensile strength.

Dr. Jeffries, in his experiments with copper at a given temperature, obtains higher strength and lower ductility in worked samples as the degree of deformation increases; with annealed samples he obtains higher strength and higher ductility as the grain size decreases. This association of high ductility with small grain size as exemplified by the quoted statement, "The elongation of annealed copper at all temperatures was less, the larger the grain size," gets me into difficulty. In these particular experiments, metal was actually annealed before test at only two temperatures, 750° and 450° C., yielding a quite coarse and a very fine structure, respectively. Other conclusions are based on predicted changes of grain size as a result of annealing during test. In the case of iron, a sample annealed at 800° C., showing a considerably coarser grain than one annealed at 750° C., gives the lower elongation at any temperature of test.

It cannot be argued that all simple metals or solid solutions, when tested below their equi-cohesive temperatures, will become more ductile as their grain size decreases, because examples of a contrary behavior are very well known. Thus, throughout, a normal annealing range of several hundred degrees ordinary cold-worked 70/30 brass increases quite as regularly in ductility as it does in grain size. Beyond a certain temperature (which varies with the composition of the metal, the amount of surface exposed, the period of annealing, and the annealing atmosphere) the ductility commences to decrease although the grain size continues to increase. I have always attributed this to general deterioration of the metal, although certain other factors, such as the relation between area of section and grain size, may be of importance.

If it is assumed that a metal remains normal in all respects during the annealing treatment and if the grain sizes in question are small enough to more or less nullify the adverse effect of directional properties (premature break at a given section because directionally weak grains preponderate in that region), should not a decrease in grain size bring about a decrease in elongation for substantially the same reasons that an increase in the

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<sup>4</sup> C. W. Bennett: "Tensile Strength of Electrolytic Copper on a Rotating Cathode." Thesis, Cornell University, 1912.

degree of deformation does? And should not this be a general property of all metals treated equivalently and tested below their equi-cohesive temperatures, so as to produce the effect of increasing reinforcement of the structure with the stiffer amorphous material as the grain size or number of inner surfaces increases? Bennett's copper, and brass at large, fit this argument. Jeffries' coarse-grained copper is not anomalous, for, to use his own phraseology, "its elongation at the lower temperatures is less than ordinary annealed copper, because it has comparatively few grains on a cross-section." Doubtless orientation had something to do with the low elongation.

There are no lack of reasons why coarse-grained structures might be expected to show impaired ductility. Such structures are usually produced by annealing at temperatures that may be regarded as high for the metal in question. So much in the way of mechanical stability depends on the grain or fragment boundaries that any cause tending to alter their nature or continuity should have a profound effect on ductility. Annealing at high temperatures may bring minute quantities of dissolved or intermixed substances into reaction with the evolution of gas, which should collect in the weakened boundaries. A little hydrogen and a little cuprous oxide would produce this effect in copper. It may actually volatilize metal from the boundaries.

Some of the experimental data reported by D. K. Crampton, in a recent thesis,<sup>5</sup> have a certain bearing on this question. In these experiments, test pieces cut from severely cold-worked 70/30 brass,  $\frac{1}{8}$  in. thick, were first annealed in duplicate sets at a given temperature. One set was then rolled to a very slight reduction, after which both were re-annealed at a temperature lower than before. Selective growth occurred in the first set and, as both had received identical heat treatment, certain conclusions as to the effect of temperature and grain size on the strength and ductility could be made.

In both sets, maximum ductility occurred after anneal at 750° C.; that is, the effect of temperature was, in general, greater than the effect of grain size. Selective growth at 750° C. produced about the same grain size (6.38 gr. per sq. mm.) as normal growth at 825° (6.08 gr. per sq. mm.) but the elongation, in 2 in., of the material annealed at the lower temperature was 84 per cent. as against 77 per cent. in the material annealed at the higher temperature. As these test pieces were annealed in air, there has, of course, been a complication of volatilization and oxidation at the surface of the metal. The ordinary relationships between mechanical properties and annealing temperatures of copper, brass, and other metals are so important that any theoretical effort that

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<sup>5</sup> D. K. Crampton: "The Influence of Abnormal Grain Growth on the Mechanical Properties of Alpha Brass." M. S. Report. Sheffield Scientific School of Yale University, 1918.

may lead to a better understanding of them may be regarded as well spent.

Some two years ago, in conversation with Dr. Jeffries, I called attention to the dissimilarity between copper and brass, in that while the former showed about the same ductility over a wide annealing range, the latter continually increased in elongation over a similar range and I found him ready with an explanation based on the amorphous theory. Using the theory in its present development, a given property—say elongation—will vary more with the grain size at room temperature in the case of brass than in the case of copper because the former is further from its equicohesive temperature than the latter; viz., crystalline and amorphous copper are more nearly alike in cohesion than crystalline and amorphous brass. Furthermore, as Dr. Jeffries has observed in the results of Mathewson and Thalheimer, there may be little difference in the grain size of copper over the annealing range in question. This appears to be rather common in the annealing of copper, at any rate changes in grain size with the temperature in the medium range of annealing are not striking as in the case of brass; which, of course, aids us to understand the test results.

In the upper part of the annealing range, the ductilities of both brass and copper diminish. In the lowest part, the ductilities again diminish; here there is perhaps room for some conflict of opinion.

I shall be greatly interested to learn whether Dr. Jeffries can agree with me that the ductility decreases as we pass from annealed metal, the grain size of which is still coarse enough to permit direct observation, into the range of worked metal where the term "fragment" may replace the term "grain," mainly because of the decreasing size of particles possessing the normal attributes of a crystal, whether they be called grains or fragments, and each surrounded by amorphous or subcrystalline material which supplies the reinforcement. The action in this case is very similar to the action originally described, wherein part of the reinforcement is due to amorphous metal along true slip planes. In either case the reinforcement raises the load that is necessary to further stretch the metal, while with additional stretch the resistance increases owing to the formation of more amorphous material, until finally the amorphous metal gives way locally and follows the line of least resistance through the metal. My interpretation, however, indicates a clear relation between ductility and grain size; very fine grain has a shorter road to travel before rupture than moderately coarse grain.

I do not mean to argue that there are no characteristic differences between a very fine-grained annealed metal and a coarse-grained metal that, by cold working, has become resolved into fragments similar in size to the grains of the annealed metal. There is certainly some form of internal strain associated with the worked metal. The two structures

must represent a different distribution and probably different quantities of amorphous material; also a different scheme of orientation of the component grains or fragments. It has been suggested that the original grain boundaries have received additional amorphous material by the action of slip in this vicinity. Doubtless they continue to receive additions during their change of shape as the bar lengthens and very likely the fragment boundaries subsequently formed contain quantities of amorphous material more or less in proportion to their age. The characteristic irregular lines that develop when severely worked metal is etched seem readily explainable in some such manner.

The elongated and thickened boundaries are strong in tension but weak under certain other forms of loading. Thus a hard-rolled brass strip mounted lengthwise in a vise and bent back and forth fails much sooner than when mounted crosswise and the fracture follows the boundaries.

In spite of the differences thus noted, I believe that the two structural types just mentioned are alike in the main essentials and that the great change in properties brought about by cold working is due chiefly to an extremely thoroughgoing mechanical refinement of grain. We have in Messrs. Bassett and Davis' paper some very carefully prepared data on grain size versus hardness in cartridge brass, which may be used to illustrate this point. Their coarsest grains count about  $8\frac{1}{2}$  per sq. mm. and correspond to a Brinell hardness of about 41, while the finest grains that could be reported with any certainty counted about 2000 per sq. mm. and raised the hardness to about 75. Beyond this, the structure could not be resolved for counting but my own examination of similar material has convinced me that grains that could count more than 1,000,000 per sq. mm. can be distinguished and these have grown out of an irresolvable environment of still greater fineness corresponding to a hardness of approximately 170. The count stands in the relation of 1:235:120,000+ and the hardness values: 1:2-:4+. From these figures it would appear that Brinell hardness is related to grain size through some exponential law:

$$\begin{aligned}\text{Brinell hardness} &= K \frac{8}{\sqrt{\text{number of grains per sq. mm.}}} \\ \text{or} &= K \frac{1}{\sqrt[4]{\text{diam. of average grain, in mm.}}}\end{aligned}$$

The value of this constant appears to be in the neighborhood of 30.

PAUL D. MERICA,\* Washington, D. C. (written discussion†).—Dr. Jeffries gives in this paper a more detailed account of his investigations on deformation, the results of which were indicated in a general way in his article on the Metallography of Tungsten,<sup>6</sup> and we are

\* U. S. Bureau of Standards.

† Received Feb. 17, 1919.

<sup>6</sup> This volume.



indebted to him for the discovery of some very interesting and curious facts. Most important is his confirmation of Beilby's experience that decrease of temperature would cause increase of ductility in copper. The possibilities of obtaining commercially valuable variations in the mechanical properties of wrought materials by working them at different temperatures are quite fascinating. But the greatest value of the experimental material of the author lies in its bearing upon the theory of plastic deformation in metals, and the author's thought and speculation along these lines is original and highly illuminating.

We are, it seems, being gradually forced, by the facts presented in these papers of Dr. Jeffries, to a more careful analysis of such phenomena as ductility, tensile strength, and brittleness in metals than has been given these simple appearing concepts in the past. It may well be found that such complex phenomena as the author describes will reduce to very simple terms when our analysis has finally been completed.

Ductility, as the author points out, must depend primarily on resistance to rupture. All metals could be deformed if stresses great enough to produce deformation were not sufficient in some cases to produce first rupture. The mechanism of deformation in metals is well known in its general features. It occurs by progressive slipping along definite planes within each grain or crystal caused by shearing stresses parallel to those planes. The shearing stress per unit area of such a plane at which slip just begins is an important physical constant of the metal, and depends probably only on the temperature and the orientation of the plane in question. After slip has occurred along any plane, the regular arrangement of several layers of atoms or molecules at this plane has been disturbed; these layers are amorphous. Further slipping along this plane will occur under the action of shearing stresses, which will overcome the resistance of viscous flow of the amorphous metal; this stress probably depends on the rate of deformation or flow as well as on the temperature. The yield point of an aggregate of grains is a most complex function of both of these two limiting stresses and of the statistical orientation of the possible slip planes with reference to the direction of the applied stress.

Although we can thus form a fairly definite picture of the mechanism of deformation, we know comparatively little about that of rupture. Where does it occur and how? Does its path lie within amorphous metal or within crystalline metal? Does it occur simultaneously or is there an initial local failure which rapidly and progressively develops into a complete fracture?

The author's results throw some light on the location of the path of rupture. The increase of ductility and tensile strength of copper at lower temperatures indicates that the breaking strength of the metal at the path of rupture increases with temperature decrease more rapidly

than the elastic limit of the crystalline metal in shear. It is not readily conceivable that the breaking strength of the crystalline metal would do this; one would rather expect a less rapid increase of breaking strength than of elastic limit. It must therefore be the amorphous metal of which the alteration in breaking strength is the determining factor.

This conclusion brings with it a rather curious corollary that although below the author's equicohesive temperature the elastic limit in shear along any crystal slip plane is less than the apparent elastic limit of the amorphous metal, the breaking strength in shear or tension of the amorphous metal must be less than that along any crystal planes other than those at which slip can occur. At the latter planes the breaking strength of crystalline metal cannot be determined as slip occurs with generation of amorphous metal before rupture.

The author's discussion of the mechanism of rupture is interesting and he points out the significance of the probable fact that rupture does not occur simultaneously. He concludes, however, that the area or intricacy of the path of rupture affects the measured breaking strength of an aggregate of grains of metal. This does not seem consistent with the first conclusion, for the breaking strength is the maximum stress that a material will bear before fracture. If fracture during the ordinary tensile test is progressive, the first local failure within the mass causes a reduction of the test load and the maximum tensile stress has been measured before rupture has fairly begun. The path of the ensuing rupture can hardly have determined the maximum stress as so measured.

The rather curious conclusion is reached that the ultimate breaking strength, at least in tension, shear, or torsion, depends on the strength of the weakest part of the metal or on the value of the highest local internal stress or on both. It may therefore be related principally to: (1) the degree of uniformity of breaking strength of individual component elements of a mass of metal, or (2) the uniformity of the local internal stresses produced by the application of an external load. In this sense it would be chiefly a statistical characteristic of the particular mass of metal in question; *i.e.*, of the orientation of its grains, etc.

This raises a question, which has been in my mind for some time. In view of the quite obvious complexity of our supposed simple mechanical concepts and characteristics of metals, why should we not revert for our fundamental investigations of deformation to the methods of Osmond and others, and use for this purpose individual crystals or grains? I believe that our progress in the interpretation of mechanical phenomena in metals will be slow until this simplification is introduced, at least until certain fundamental facts are established.

It is difficult to find an explanation of the fact that although the tensile strength of both copper and iron increases as the temperature decreases from 300° C., the ductility of copper increases but the ductility

of iron decreases. The author attributes this decrease of ductility of iron to the fact that internal stresses set up between the amorphous and the crystalline metal weaken the metal along the path of rupture. Yet the tensile strength does actually increase very rapidly and shows no indication of this weakening effect.

The author's suggestion as to the cause of brittleness in tungsten at low temperatures as being due to internal stresses set up between the amorphous intercrystalline metal and the crystalline grains is interesting. If the rate of contraction on cooling of the amorphous metal is less than that of the crystalline metal below the equicohesive temperature, as the author believes, the system of stresses set up in a structure consisting of continuous thin walls of amorphous metal inclosing grains of crystalline metal on cooling would consist of a negative hydrostatic pressure within the grains, and within the amorphous envelopes tensional stresses normal to the envelopes and compression in all directions in their plane. Secondary stresses somewhat similar to shearing stresses would be produced at the boundary of envelopes and grains tending to strip envelope from grain, and thus to produce a weakening of the whole structure.

S. L. HOYT, \* Minneapolis, Minn.—At present, there seem to be two schools in metallography, one which has adopted the amorphous-phase theory for the explanation of the effect of cold work, mechanical deformation, etc., and one which is not ready at present to adopt this theory. It seems to me that this theory can at least be used as a convenient means of explaining various facts that are demonstrable in the laboratory but the presence of this amorphous material, through our lack of experimental means, we are unable to prove. That we have failed in this direction should not necessarily hinder us from using a theory that is as workable as this.

I understand Dr. Jeffries to say that fracture occurs between the crystals at room temperature. According to the amorphous-phase theory, as originally expounded by Dr. Rosenhain, if I remember correctly, fracture occurs across the crystal. Now, is it true, assuming that we have amorphous material, that fracture always occurs in the amorphous material, whether that amorphous material is between the grains or in the grains along the slip planes? We have heard a very great deal lately about allotropy in iron, although those discussions had to do with the beta and gamma phases rather than the possible presence of low-temperature allotropy. So far as my interpretation of the curves given by Dr. Jeffries, showing the variation of tensile strength, elongation, and reduction at variable temperature, enables me to predict, the tensile-strength curve shows that the change takes place, speaking in degrees

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Centigrade absolute, from  $300^{\circ}$  to  $500^{\circ}$ , the curve for elongation shows the change to take place from  $300^{\circ}$  to  $400^{\circ}$ ; and the curve reduction of area shows that it takes place from  $300^{\circ}$  to  $600^{\circ}$ . In other words, there seems to be a lack of agreement as to the temperature range over which this allotropic transformation has taken place, depending on whether the determination is that of tensile strength, reduction of area, or elongation.

On page 552, the first sentence in the first paragraph reads: "The rate of transformation from one allotrope to another should depend on the cohesion of the metal at the temperature at which the change should take place." This brings up the question of the rate of change of one allotropic modification into another allotropic modification, and the variation of that rate of change with temperature; and Dr. Jeffries brings up the question as to whether that change or rate of change depends on cohesion of the material. If it does—as we accept, of course, that as the temperature lowers the cohesion increases—we must assume a similar and corresponding increase in the rate of transformation of the one allotrope into the other allotrope. Prof. Tammann and collaborators have determined experimentally the rate of crystallization of various materials from their liquids; if we accept this work, we will see that the rate of generation of crystals of the new substance, as well as the linear velocity of crystallization or deposition about these crystal centers, is a function that varies in quite an irregular manner with the temperature. At least the variation in this function in temperature is quite different from the variation in cohesion. Prof. Tammann has shown that, at the equilibrium temperature, that is the temperature at which the two allotropes could coexist in stable equilibrium, the rate of formation of crystal centers of the new substance is practically zero. As the temperature drops to below the equilibrium temperature, there is a slow and then a very rapid increase in the rate of formation of the crystal nuclei; but very soon a maximum is reached, below which temperature the rate of formation of crystal nuclei takes an equally sudden drop. In other words, the rate of variation of the formation of crystal nuclei is entirely different with dropping temperature from that of the cohesion. If we are to consider the rate of transformation of the allotrope into another, we must take into consideration the work that Prof. Tammann has done.

One or two other points occur to me in this connection. If the rate of transformation varies directly with the rate of increase in cohesion, we would expect that at a very low temperature we would get a marked increase in the velocity of transformation. Accepting the view advanced by Prof. Tammann, it would seem that, if temperature has such an effect and if we get a transformation at low temperatures that we do not get at the higher temperatures, we must look to some other cause as the explanation of it. It is true there is a transformation from austenite,

that is, the high-temperature modification of steel into martensite at a temperature slightly above that of liquid air, and that point has been advanced by Dr. Hanemann, as a confirmation of the view Dr. Jeffries advances here; that is, that the further we get from the true equilibrium temperature the greater will be the tendency to change. Dr. Guertler, in discussing this theory, pointed out that it was not at all necessary, in fact it was quite contrary to the generally accepted belief, for the rate of transformation to increase with temperature. I believe that these points have an important bearing on the effect of deformation of iron in particular because, as Dr. Jeffries says, there is a possibility of there being a low-temperature allotrope.

I should also like to ask Dr. Jeffries if he performed his experiments on both rising and dropping temperature. When we investigate allotropic transformations, we usually investigate them in both directions; the transformation should take place in one direction on heating and in a reverse direction on cooling. If there is a true allotropic transformation, it is quite necessary that there be hysteresis; if these transformations take place in the reverse direction on cooling from that in which they occur on heating, but the temperature at the beginning of the transformation is lower on cooling than on heating, there will be a strong foundation for the assumption of allotropy. But if the tensile strength, reduction of area, etc. of iron, at some certain temperature, for example  $200^{\circ}\text{C}.$ , are the same whether approached from below or above that temperature, we will have a temperature effect as distinguished from an allotropic effect and the change in properties that takes place over this range cannot be considered as due to allotropy or due to a phasial transformation. I assume that Dr. Jeffries is considering here a definite physical transformation, when he speaks of the variation in properties between  $0^{\circ}$  and  $200^{\circ}$  to  $300^{\circ}$ .

ZAY JEFFRIES, Cleveland, Ohio (author's reply to discussion\*).—The discussions by Mathewson, Merica, and Hoyt reflect the same general view as that obtaining at present on evolution, namely, every one now believes in the general theory but there is much speculation regarding the precise steps leading up to the development of the higher forms of animal life. So too, the amorphous metal hypothesis of Beilby, Bengough, and Rosenhain and Ewen is now very generally accepted as the best working theory but there is and will continue to be much speculation as to the nature of contact between amorphous and crystalline metal, the thickness of the amorphous films and their exact distribution, especially in cold-worked metals.

For several years I have had the opinion that the amorphous films between equiaxed grains were thicker in fine-grained than in coarse-grained samples and that the minimum thickness must be greater than

100 atoms. There are several reasons for the above beliefs and I hope to have some experimental evidence soon. An amorphous film one atom thick (as would be expected if surface tension forces alone predominated) is entirely inadequate to account for certain properties.

With reference to Dr. Mathewson's discussion on the relation between grain size and elongation, I might refer to the statement near the top of page 541, "It would not be surprising if all metals at any given temperature would have maximum elongation and reduction of area with a certain small grain size, and either smaller or larger grains might produce less elongation or less reduction of area." I had in mind here that the limiting condition with decrease in grain size would theoretically be reached only when the whole mass was amorphous. Certainly at low temperatures elongation would begin decreasing long before the metal reached this condition. I agree with Dr. Mathewson, in the main, on the relations between cold-worked metals and change of grain size in annealed metals. It is obvious that the shapes of grain fragments produced by deformation will be different from the shapes of grains in annealed metals and the shapes determine the *position* of the *A* metal, and the size and shape of grain or fragment determines the *quantity* of *A* metal. Several observations lead me to believe that the thickness of the *A* metal films at slip planes in deformed metal is greater than the thickness of the films between grains of annealed metal. If this is true the reinforcement would be greater in the former case.

The idea has been expressed, in a number of places recently, that cold-worked metals regain ductility on heating before recrystallization. The writer has never believed this and it is interesting to note that Dr. Mathewson does not. It may be true, however, that ductility is regained before visible recrystallization. It is highly probable that recrystallization has taken place whether or not evidence can be seen under the microscope and that restored ductility is a result of recrystallization. Dr. Mathewson's formula relating Brinell hardness to grain size is extremely interesting. It would be as interesting to compare the relations between the tensile properties and grain size.

With reference to Dr. Merica's discussion, the writer mentioned in paragraph 7, of the Summary, that the maximum breaking load would be a balance between the two tendencies: rupture by degrees, which tends to lessen the maximum strength, and the increased path of rupture caused by deformation in ductile metals, which tends to increase it.

The matter of per cent. elongation in iron in liquid air is very perplexing. One annealed sample was stretched at room temperature 10 per cent. and then cooled to liquid air and it stretched 6 per cent. more before breaking. It should have elongated about 25 per cent. at room temperature and about 11 per cent. in liquid air if it had not been elongated any at room temperature. This and other tests indicate that cold-worked

iron has a tendency to be brittle in liquid air. We are not sure that iron at room temperature is completely in one allotropic condition. More experimental work is urgently needed to throw further light on this subject. Dr. Merica's suggestion that more experimental work be done on single crystals is most timely.

Prof. Hoyt's ideas on allotropic transformation do not differ markedly from mine. It is probable that the force tending to produce the allotropic change is zero at temperatures higher than that at which it is perfectly stable on heating, and that the force tending to produce the change on cooling increases with decrease in temperature. There will be a temperature not far below that at which the change should take place on cooling, which will produce the maximum velocity of transformation. That this is true is shown by many experiments on steel, water, silver, and silica. Water and silver may be undercooled (silver as much as  $70^{\circ}$  and water  $17^{\circ}$  C.) before the change from the liquid to the solid state begins, if the materials in the liquid states are maintained in a very quiet state; then solidification takes place more rapidly than if undercooling had not taken place. The most rapid transformation in carbon steels occurs at a temperature about  $100^{\circ}$  lower than that at which it would occur on slow cooling.

Austenitic manganese steel will apparently remain in that condition at room temperature indefinitely. At  $675^{\circ}$  C., however, the change from austenite to martensite occurs after a few hours and if the austenite is deformed at  $400^{\circ}$  C. the transformation proceeds far enough in a few minutes to make the steel magnetic. This shows that mechanical disturbance of the atoms by deformation favors this transformation.

Silica and glass at room temperature will remain in the amorphous state indefinitely; but when heated to high temperatures for prolonged periods they crystallize. It therefore seems that the mechanical cohesion resists allotropic transformation. The change from austenite to martensite in high-carbon steels, brought about by cooling in liquid air, does not fit in with the above ideas but it is true that we know less about this change than the others cited above.

So far as I know, no work has been done on the relative positions of the maxima and minima points of the tensile properties of iron on both rising and falling temperatures. This line of investigation should prove interesting.

## Die Castings and Their Application to the War Program

BY CHARLES PACK,\* BROOKLYN, N. Y.

(New York Meeting, February, 1919)

DIE castings may be defined as metal castings made by forcing molten metal, under pressure, into a metallic mold or die. It is necessary to keep this definition in mind to avoid confusing this process with other permanent-mold casting processes. The fundamental principles of the process have been known and practised many years. The simplest application is embodied in the modern linotype machine in which molten metal (usually tin-lead alloy) is forced under pressure into a metallic mold. The pressure is derived from a piston and cylinder immersed in the molten metal. Progress in the art of die casting may conveniently be divided into three groups: Machine for imparting pressure to the metal, material for the die or mold, casting alloys.

The problem of delivering molten metal under pressure into a die is comparatively simple, when dealing with low-fusing-point alloys, as the alloys of lead and tin, but it is much more complicated when dealing with metals of higher fusing points, such as the alloys of zinc, aluminum, and copper. Although the art of die casting is comparatively new and, to a large extent, unknown, the records of the patent office are replete with patents on the subject.

Fig. 1 shows the Underwood machine patented in 1902; this is probably one of the first machines designed for the production of commercial die castings. The relation of this machine to the linotype casting machine is clearly apparent. A cylinder and piston are immersed in the molten metal, the application of power to this piston forcing the molten metal, under pressure, into the mold or die. The Doehler machine, Fig. 2, patented in 1907, is based on the same general principles. This machine is used to a large extent at the present time, throughout the United States, for the production of zinc, tin, and lead alloy die castings.

In the machine shown in Fig. 3, patented by Doehler in 1910, compressed air is used for forcing the metal into the die. In Fig. 4 is shown another of this type of machine. Here compressed air is applied to the surface of the molten metal to force it into the die.

In a machine patented by Chandler in 1914, shown in Fig. 5, the

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\* Chief chemist, Doehler Die Casting Co.



principle of the internal-combustion engine is applied for exerting pressure on the molten metal. A charge of gasoline vapor and air is injected into the melting chamber, the explosion of which forces the metal into the die. The writer has never heard of this machine being used on a commercial basis, but it is mentioned to show the various means suggested for forcing molten metal into a die.

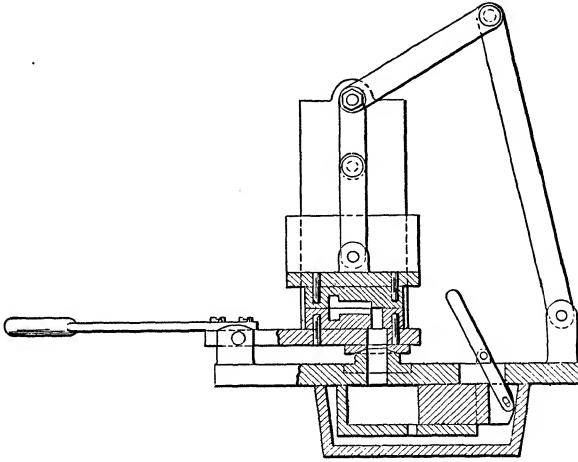


FIG. 1.—UNDERWOOD DIE-CASTING MACHINE.

#### METHODS USED TO AVOID BLOW-HOLES

The fact that die castings are made under pressure would suggest, on first thought, dense and homogeneous castings; this impression, however, is not in accord with actual practice. On fracture, the pressure die casting will be found to consist of a dense closely grained outer stratum and a porous inner stratum. Blow-holes of varying size may be expected in the center of the die casting, particularly through heavy sections. Many machines have been designed with the primary object of overcoming this difficulty and producing solid die castings.

Fig. 6 shows an air-operated die-casting machine with the die inclosed in a vacuum chamber. The inventor evidently assumed that the only cause for blow-holes in the casting was the presence of air in the die. In Fig. 7 is shown another die-casting machine in which the vacuum principle is applied; here the vacuum is applied directly to the die.

The production of die castings free from blow-holes has been the most serious problem confronting die-casting manufacturers. At various times it has been stated that processes capable of producing solid and homogeneous die castings have been developed. If all blow-holes in die castings were caused by air coming in contact with metal, the vacuum process would deserve consideration. That the presence of blow-holes

in some die castings are due to other and more serious causes, the writer will endeavor to prove.

In Fig. 8 is shown a cross-section of a casting that can be gated at *A* or *B*; in the best foundry practice the gate *A* would probably be used. The first metal that goes into the die will chill around the inner walls and take the form shown in the shaded portion. The gate may then be

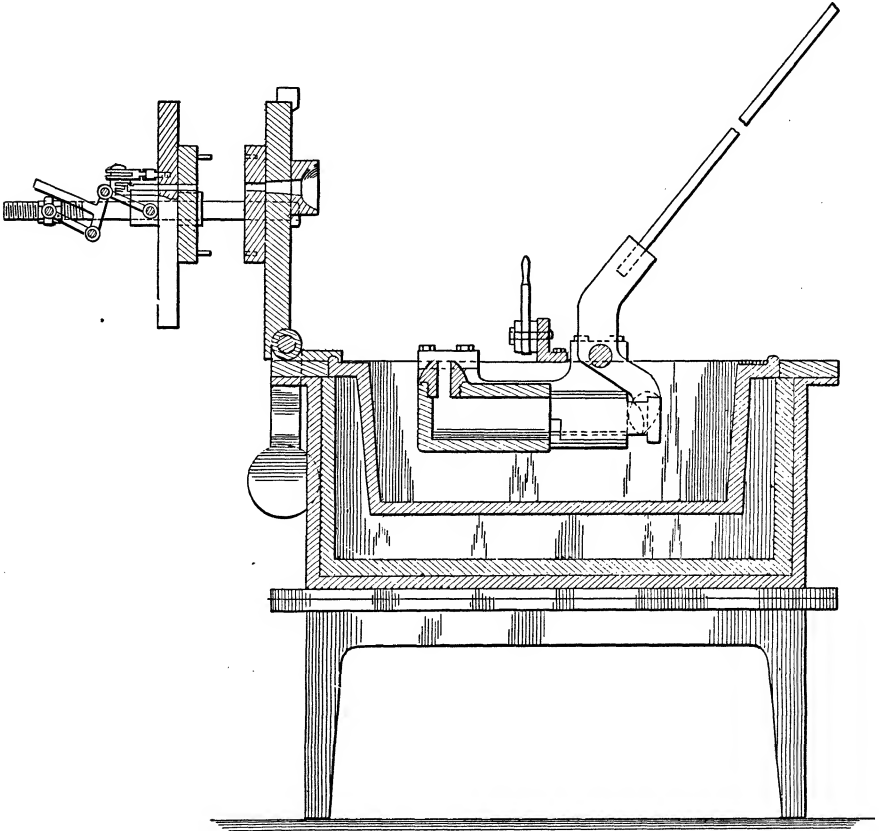


FIG. 2.—EARLY TYPE OF DOEHLER MACHINE.

come chilled before the inner portion has been filled; this will cause blow-holes that no vacuum will eliminate. A similar effect will be produced if the metal was too cold at the time of casting. The writer has produced castings having only an outer shell, similar to that shown in Fig. 8, by limiting the amount of metal injected into the die to a quantity less than that required to make the casting. A similar result may be obtained by running the metal so cold that it will chill the thinner sections of the casting before the heavier sections are completely filled. Lack of pressure will produce the same result.

Blow-holes in die castings may also be caused by the phenomenon that we sometimes call "piping." Makers of rolling-mill ingots have often been confronted with this problem. In Fig. 9 is shown cross-section of another casting gated at A. The metal flowing into the die at A

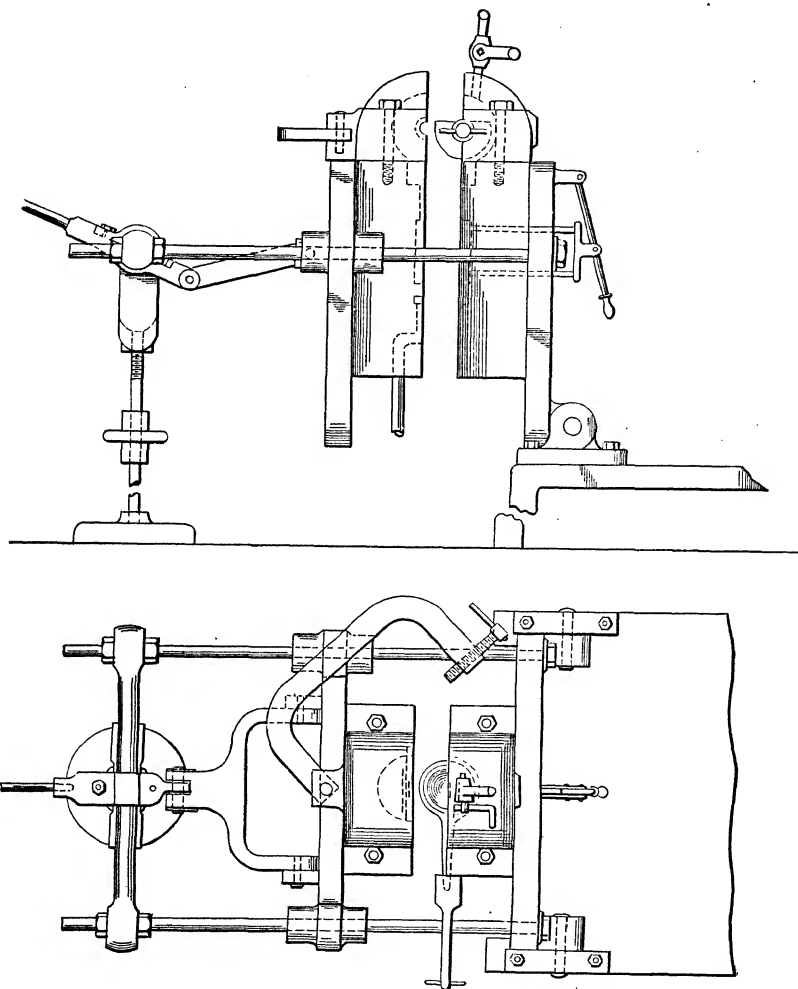


FIG. 3.—DOEHLER COMPRESSED-AIR MACHINE.

will fill the entire mold cavity, assuming all casting conditions to be ideal, but the metal in the thin section adjoining A will chill before the heavier section so that, the chilling being from the outside, a shrinkage hole will be left in the center. Here again no advantage can be gained by the use of the vacuum system.

## DIES

In the manufacture of die castings from zinc, tin, and lead alloys, dies made from low-carbon machine steel last almost indefinitely and answer every purpose. In the first attempts to die-cast aluminum, the problem of obtaining a suitable die material presented serious difficulties, which were described by the writer in a paper read before the

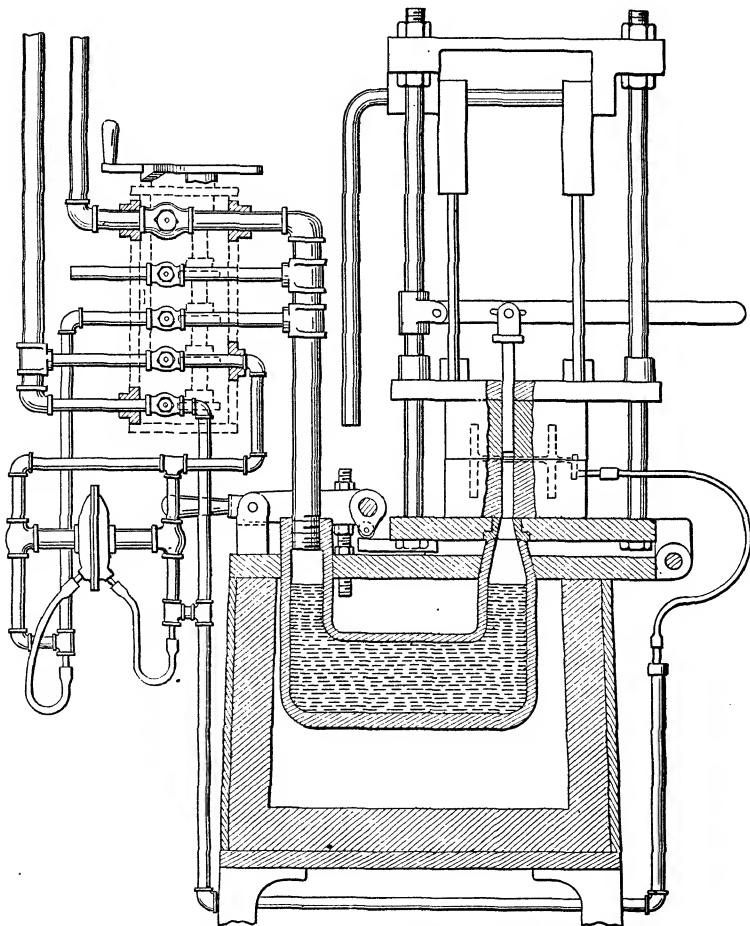


FIG. 4.—ANOTHER TYPE OF COMPRESSED-AIR MACHINE.

American Institute of Metals in 1915. This problem, however, has been solved by the use of various alloy steels making possible the commercial die casting of aluminum and its alloys, which constitutes the greater part of the die-casting industry of today. The proper gating and venting of these dies are problems that arise daily and on the solution of these problems depends the success or failure of the process.

## ALLOYS

In a paper read before the American Institute of Metals in 1914, the writer described the various types of zinc, tin, and lead alloys used in the die-casting process. The application of these alloys and their limitations were also pointed out. At that time the die casting of aluminum and its alloys was barely beyond the experimental stage. During the

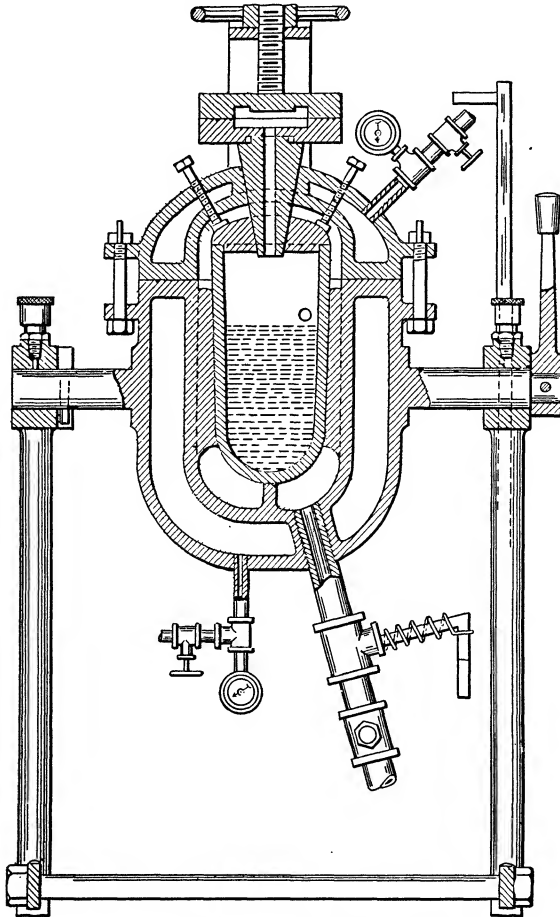


FIG. 5.—CHANDLER DIE-CASTING MACHINE.

past 4 years, the most important advance in the art of die casting has been made in the perfection of the process for die-casting aluminum and its alloys. The importance of this achievement as an aid to winning the war is best demonstrated by the fact that at least 95 per cent. of the die-cast parts used directly or indirectly as materials of war were made from an aluminum-base alloy. Of these castings, only a very small percentage could have been produced successfully in 1914.

Investigations of the casting properties of metals and alloys in the past have been generally limited to sand castings; few data are available as to the casting properties of metals or alloys in metallic molds. Just what constitutes a good die-casting alloy is a subject of unusual interest. A few of the important requirements, outside of the usual physical properties demanded of alloys, are:

*Melting Point.*—The successful die-casting machine in every instance is constructed of iron, in one form or another. The melting point of the alloy must be such that it will melt readily in an iron pot.

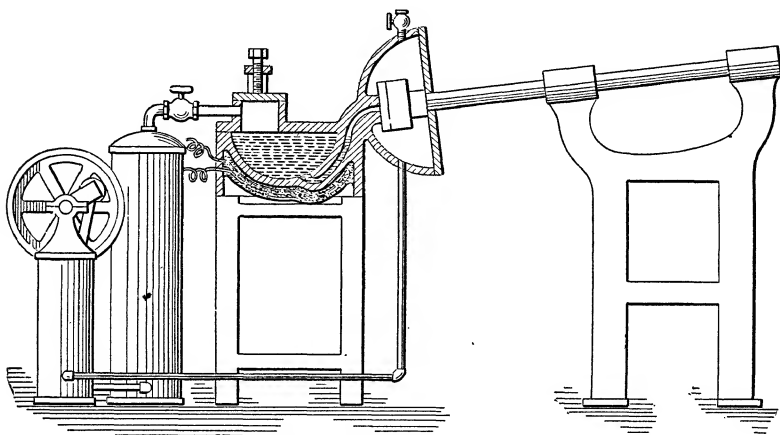


FIG. 6.—MACHINE WITH DIE ENCLOSED IN VACUUM CHAMBER.

*Solvent Action.*—The solvent action of the alloy on iron must not be too great. Molten aluminum dissolves iron very rapidly and analyses of aluminum die castings on the market will show an iron content of from 1 to 3 per cent., due to the solvent action. Fortunately, there is no serious objection to the presence of iron in aluminum casting alloys. Should the aluminum absorb much above 3 per cent. iron, the melting point becomes too high and the alloy becomes viscous and unsuitable for making castings.

*Elongation.*—The elongation of the metal is of vital importance in determining the die-casting properties of an alloy. Not only is it desirable to know the elongation of the alloy when cold, but it is of greater importance to determine the elongation at various temperatures ranging from the melting point of the alloy down to normal temperature. The reason for this becomes apparent when the physical phenomena of the die-casting process are considered. Let us assume that a ring 12 in. (30.48 cm.) in diameter is to be die-cast in a metallic mold around a metallic core. As the molten metal strikes the mold it solidifies. Here a change of state occurs that is accompanied by

a reduction in volume, commonly termed shrinkage. Unlike a sand core, the metallic core is not compressible and retains its original size and form so that the shrinkage of the metal is converted into a stretching action on the solidified casting. If the elongation of the alloy at that temperature is not high enough to withstand this stress the casting will crack. In the usual die-casting practice it is not

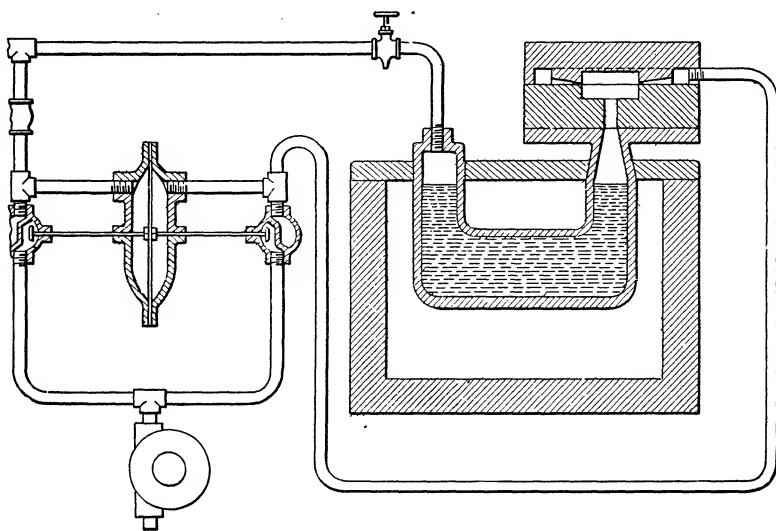


FIG. 7.—DIE-CASTING MACHINE WITH VACUUM APPLIED DIRECTLY TO DIE.

practical to remove the casting from the die at the solidification temperature of the alloy. For example, the solidification temperature of the aluminum-copper alloys used in the die-casting process is approximately 1150° F. (621° C.). It has not been found practical to run the casting dies above a temperature of 500° F. (260° C.), which means that the castings are withdrawn from the dies at that temperature. It follows that the casting is subjected to another stretching stress after the casting has solidified, due to the contraction in volume that must occur when a casting is cooled from a temperature of 1150° F. to 500° F.

The writer has been unable to find any reliable method for determining quantitatively the elongation of alloys at various temperatures. Many methods have been suggested but they have proved of doubtful value. The simplest way is to use the old "try-and-see" method. To test the alloy, a casting is made in a die having a comparatively large core and thin wall. If the alloy can stand the casting stress, a perfect casting will be obtained, otherwise the casting will show bad cracks. Only a comparative result is obtained, but for everyday control it answers the purpose. However, a simple and reliable method for deter-

mining quantitatively the elongation of metals and alloys at various temperatures would prove of enormous value to all metallurgists engaged in the various phases of metal-casting research.

It is interesting to note that the elongation of a metal or alloy at normal temperatures is no indication as to the properties of that metal or alloy at higher temperatures. The writer has found many cases where

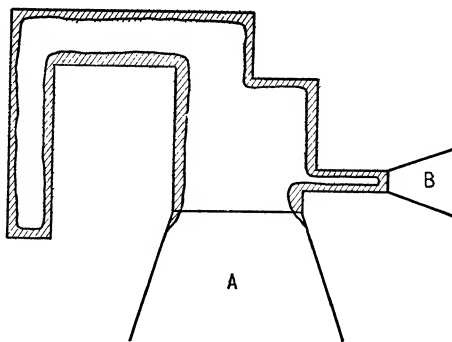


FIG. 8.—CROSS-SECTION OF CASTING WITH TWO GATING POSITIONS.

an alloy showing little or no elongation at normal temperatures shows a high elongation at higher temperatures. The alloys of aluminum and copper may serve to illustrate this point. It is well known that the addition of copper to aluminum reduces the elongation of the aluminum alloy. An aluminum alloy containing 12 per cent. copper will show less elongation than an alloy containing only 6 per cent. copper when tested at normal temperatures. Nevertheless, the 12 per cent. copper alloy has

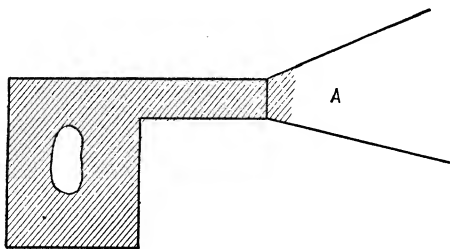


FIG. 9.—DIE CASTING CONTAINING SHRINKAGE HOLE.

a greater elongation at higher temperatures than the 6 per cent. alloy and consequently the 12 per cent. alloy is better able to withstand the casting stresses to which it is subjected in the die-casting process.

In the early days of the die-casting industry, alloys were compounded indiscriminately and little or no consideration was given to the metallurgical principles involved. The manufacturer in many instances



knew much more about machinery than about metals. The result was that there were put on the market die castings made from alloys that deteriorated rapidly and created a prejudice among engineers against the use of these castings. That this prejudice was in part justified must be admitted; nevertheless, the modern die-casting plant is equipped with physical and chemical testing laboratories and the die-casting practice of today bears no relation to that of 5 years ago.

### DIE CASTINGS MADE FOR WAR PURPOSES

Die castings have had their most severe test during the past 2 years, during which time most of the die castings manufactured were used directly, or indirectly, in the Government's war program. Here is a partial list of the application of die castings for this purpose.

Gas masks, breather tubes and other metal parts.

Lewis machine guns, 100 die-cast parts to every gun.

Browning machine guns, four of the most vital parts.

Naval and army binoculars, the entire housing.

Army truck, tank, and airplane die-cast parts include parts of ignition system, carburetor gasoline-regulating devices, steering-wheel accessories, ball-bearing cages, bearings, speed indicators, etc.

Pistol, complete signal pistol.

Submersible bombs, some designs contained as many as 10 die-cast parts.

Hand and rifle grenades, every grenade manufactured in this country contained one or more die castings.

Trench mortar shells, plugs die cast.

Airplane drop bombs, one or more die-cast parts.

Surgical instruments, including hair clippers, respiratory devices, etc.

In many instances, die-cast parts were used where the failure of the part would result in serious loss of life. The fact that not one failure of a die casting has been reported must continue to be a source of deep satisfaction to the modern die-casting manufacturer.

### DISCUSSION

JESSE L. JONES,\* Pittsburgh, Pa. (written discussion†).—As the die-casting process is so very different in character from the process of making sand molds, it is often considered that entirely different methods of pouring, gating, venting, etc. should be used. There is really, however, no essential difference in the making of die castings and sand castings, and similar methods should be used in each process.

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\* Metallurgist, Westinghouse Elec. & Mfg. Co.

† Received Feb. 13, 1919.

In the matter of gating, the metal should be introduced at a central point, as nearly as possible, so that it will have an equal distance to flow from the point of entrance to the most distant points of the mold. This will cause a uniform rate of congealing of the casting and prevent streaks and cold shuts.

The venting should be done in such a way that a free exit of the air in the molds can occur. It should be less difficult to take care of the venting of a die-casting mold than to vent an ordinary sand mold. In the case of a sand mold, the venting is left, to a considerable extent, to the judgment of the molder, and it is possible that no two molds will be vented in a similar manner. Further than this, after a vent has been made in a sand mold, it may become clogged by the sand and for this reason be inefficient. There is also quite a variation in the pressure of the metal entering a sand mold, due to varying heights of the pouring gate. In a die-casting mold these factors can be controlled more closely. The vents should be so disposed that the air in the mold compressed by the entrance of the metal may be able to leave the mold at a uniform rate and by exits located symmetrically. These exits are made quite small on molds at first, as they can be easily enlarged if necessary, and they are tapered so that a free flow is guaranteed without danger of the metal spurting out from the die. Uniformity of pressure of the metal as it enters the mold is very important and, as the pressure is mechanically applied, its control is not difficult.

It is true that a sand mold has much less of a chilling effect on the metal poured into it than has the die-casting mold, but, outside of this point, there is a great similarity between the die-casting process and the process of making castings in sand molds. It is suggested that much would be gained by regarding these two processes as not essentially different.

## Metallography of Tungsten

BY ZAY JEFFRIES,\* B. S., MET. E., CLEVELAND, OHIO

(Colorado Meeting, September, 1918)

TUNGSTEN has the highest melting point of all the known metals, namely 3350° C.; it is one of the hardest of the metals; it has the highest equiaxing or recrystallization temperature after strain hardening, of any pure metal known. It is particularly distinguished because, when composed of small equiaxed grains, it is extremely brittle and fragile at room temperature, and when possessing a fibrous structure it may be ductile and pliable at room temperature. The common ductile metals act in exactly the opposite manner in this respect.

The present paper will include a brief note regarding the manufacture of wrought and ductile tungsten. The metallography of wrought and ductile tungsten in the various stages of manufacture will be considered more or less in detail. The general relationships between the properties of tungsten and other metals will also be considered.

A discussion will be given explaining why fibrous tungsten is ductile at room temperature, even though past experience with other metals would indicate that it should be brittle. Finally, a brief note regarding some new fundamental metallographic propositions relating to all metals will be given.

### MANUFACTURE OF WROUGHT AND DUCTILE TUNGSTEN

Up to about 10 years ago, it had not been possible to work tungsten mechanically. About the year 1908, Dr. William D. Coolidge, of the General Electric Co., produced ductile tungsten from what was then supposed to be an inherently brittle metal and which, in fact, was inherently brittle in its normal state, that is, when composed of equiaxed grains. Since that time many products, chief among which are the filaments for electric incandescent lamps, have been made from wrought and ductile tungsten.

Tungsten is produced chiefly from the minerals wolframite and scheelite.<sup>1-8</sup> The ores may be reduced in many ways. For example, the ore may be fused with alkali carbonates and the fusion dissolved in water; sodium tungstate is formed, which is soluble in water. This

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\* Director of Research, Aluminum Castings Co.

<sup>1-8</sup> References are to bibliography at end of paper.

may be changed to tungstic oxide,  $\text{WO}_3$ , by the addition of an acid to the sodium tungstate liquor.  $\text{WO}_3$ , a yellow precipitate, is separated by filtering. The tungsten oxide may then be purified to any desired degree by dissolving it in ammonia and then reprecipitating by the addition of acid, followed by filtering and washing.

For the manufacture of most of the wrought tungsten products, the tungsten oxide is treated as follows: after drying, sufficient thorium nitrate solution is added to make 0.75 per cent. thoria ( $\text{ThO}_2$ ) in the oxide. The tungsten oxide is made into a batter with the thorium nitrate solution, additional water being added if necessary, after which it is thoroughly mixed. The mixture is then dried and placed in either a fire-clay or a silica crucible and heated to about  $1100^\circ \text{C}$ . for a period usually in excess of 1 hr. This is called the firing operation, and it has for its purpose the agglomerating of the extremely fine particles of tungsten oxide into larger, or coarser-grained particles. When the firing is done in a fire-clay crucible, some of the crucible material is dissolved by the oxide, and produces a beneficial effect when the metal is to be used for incandescent lamp filaments.

The fired oxide is then reduced by hydrogen to tungsten metal powder, at a temperature in the neighborhood of  $1000^\circ \text{C}$ . The tungsten powder thus produced is relatively coarse grained when compared to the tungsten powder from which the sintered tungsten filaments were made before the advent of drawn tungsten wire.

The tungsten powder is pressed dry, into any desirable shape or size. A common size and shape consists of a rod about 16 in. (40 cm.) long having a cross-section about  $\frac{3}{8}$  in. (9.5 mm.) square. The pressure on such a rod must be applied from the side of the mold and not from the end, else the resulting tungsten ingot will not be workable. The pressed tungsten slug is so fragile that it can be moved only by sliding it along a surface. It is moved in this manner onto a platform of tungsten, nickel, or other suitable material having a high melting point, and while still on the platform is inserted into an electric furnace of the tube type, which is heated to about  $1200^\circ$  or  $1300^\circ \text{C}$ ., in which an atmosphere of hydrogen is maintained. This is called the baking operation. After baking, the tungsten slug may be handled without danger of breaking. At this stage, it is porous, containing about 40 per cent. voids.

The baked tungsten slug is then clamped into two electrodes, arranged in a vertical position, the bottom one of which floats in a bath of mercury cooled by a water jacket. This arrangement is necessary, because in the operations which follow, the tungsten slug shrinks, usually about 15 per cent. in length, and the mercury well must accommodate this shrinkage and still maintain electrical contact with the lower electrode. After the slug is securely clamped in the electrodes, a gas-tight housing is lowered around it and a current of hydrogen is

passed through this housing. Electric current is then passed through the slug, thus heating it to an extremely high temperature. For most purposes, a temperature of about 3000° to 3200° C. is used, and this temperature is maintained for 10 or 15 min. This may be called the sintering operation.

The sintered tungsten ingot is substantially free from voids, having a density of about 18. The two ends which were clamped in the electrodes will not have been heated to a sufficiently high temperature to cause sintering, so they are broken off and then the ingot is ready for the mechanical working process.

The working operation is begun in swaging machines,<sup>10-16</sup> which operate the swaging dies. A pair of swaging dies consists of two similar blocks of high-speed steel, each block containing half the desired opening. The openings are circular in cross-section. The side of the steel block containing half the desired opening is called its "face." These two half dies are placed in the swaging machine face to face, sufficiently loose so that the swaging blow can be struck, when the die halves move away from and toward each other. The machine head rotates and the dies rotate with it. Centrifugal force moves the die halves apart and rollers in the head of the machine push them together again. The dies move backward and forward several thousand times per minute. The faces of these dies must be made in a special manner for the swaging of tungsten. The portions which come in contact with the metal to be worked must be very much shorter than when swaging the ordinary metals. These are called short-faced dies. They are made of high-speed steel because they are to be used to swage hot metal.

For beginning the swaging operation on a tungsten ingot having a square or rectangular cross-section, a pair of swaging dies is selected which will just round off the corners of the ingot. The ingot is put into an electric furnace, usually consisting of an alundum tube wound with tungsten or molybdenum wire, in which an atmosphere of hydrogen is maintained. The temperature of this furnace may be as high as 1700° C. but it is more frequently in the neighborhood of 1600° C. When the ingot has attained the temperature of the furnace, one end is grasped with a pair of tongs and the other end is quickly inserted into the swaging machine. It is not possible to swage the whole length of the rod in this manner, so the ends of the rod are reversed and it is again heated, after which the unswaged end is inserted into the swaging machine. A pair of swaging dies having a somewhat smaller opening than the first pair of dies used is now put into the machine; the ingot is heated again and the swaging operations continued as before. Four or five dies are required to make a round rod from a square or rectangular ingot.

When the tungsten rod is sufficiently elongated to enable the end which first passes through the swaging machine to be gripped from the

exit side, only one heating for each pair of swaging dies is necessary. As the degree of work increases on the rod, the temperature is gradually decreased. When the working temperature has been reduced to about  $1300^{\circ}\text{C.}$ , gas may be used to heat the metal preparatory to swaging. The temperature may be decreased in steps, that is, there may be a certain temperature maintained for a half dozen or more dies and then the temperature lowered for the next half dozen dies. These conditions will depend on the properties desired in the swaged tungsten rod. The swaging operation may begin on tungsten ingots of any size, but a common size is  $\frac{1}{4}$  to  $\frac{3}{8}$  in. square (6.35 to 9.5 mm.), and 12 to 14 in. long (30.48 to 35.56 cm.).

If it is desired to produce wrought tungsten rods more than 0.030 in. (0.76 mm.) diameter, all of the work is done by swaging. If, however, it is desired to produce tungsten wires smaller than 0.030 in. diameter, the swaging operation is resorted to only until a size of 0.030 in. or thereabouts is reached. The temperature of swaging will have been reduced as the size of the rod decreased so that the 0.030 in. rod would be swaged at a temperature of  $1000^{\circ}$  or  $1100^{\circ}\text{C.}$

When the tungsten ingot is removed from the furnace previous to swaging, the temperature corresponds to a brilliant white heat. Tungsten oxide has not formed in the furnace because of the hydrogen atmosphere, but as soon as the ingot is removed from the hydrogen atmosphere, its temperature is such that tungsten oxide forms very rapidly, volatilizes and condenses as a white fume at some distance above the rod itself.

The tungsten ingots may also be reduced by rolling. High-speed rolls are employed and the ingot is heated to  $1500^{\circ}\text{C.}$  before each pass. Rolls are used for flat pieces and swaging machines for round sections.

The sintered tungsten ingot is as brittle as glass and extremely fragile. It is composed of equiaxed grains such as may be seen in Fig. 1, which represents the structure of a sintered tungsten ingot. When the swaging has been continued until the rectangular cross-section of the ingot has been changed to circular, the structure has been changed to that shown in Fig. 2. With still further swaging, when the tungsten rod has reached a diameter of 0.125 in. (3.2 mm.), its structure is changed to that shown in Fig. 3. At a size of 0.082 in. (2.08 mm.) its structure has changed to one corresponding to that shown in Fig. 4, while Fig. 5 represents the structure when the tungsten rod has been swaged to a diameter of 0.030 in. (0.76 mm.).

It will be seen from these micrographs that the equiaxed grains composing the sintered tungsten ingot have been progressively elongated during the swaging operations into fibers of metallic tungsten. The 0.030-in. swaged rod shown in Fig. 5 is ductile cold. It can be bent cold or it can be drawn cold.

While the tensile strength of the sintered tungsten ingot from which

this 0.030-in. swaged tungsten rod was reduced was only 18,000 lb. per square inch, the tensile strength of the rod was 215,000 lb. per square inch.

The sintered tungsten ingot was absolutely brittle, but the 0.030-in. swaged tungsten rod has an elongation of about 4 per cent. in 2 in. and a reduction in area at the point of fracture of about 28 per cent.

When it is desired to produce wires smaller than 0.030 in. diameter, a hot drawing process is resorted to. Diamond dies are usually employed for this purpose. The lubrication used, both for the swaging operations on the smaller rods and in the drawing process, is aquadag. The wire to be drawn is first passed through an aquadag mixture which is in the form of a thin paste, and then through a gas flame or other heating medium which heats the wire to the desired drawing temperature and also bakes on it a coating of the lubricant. The dies vary in size by small steps; that is, the diameter of the opening in one die will be only about 6 per cent. greater than that in the next following die.

The temperature at the beginning of the drawing may be in the neighborhood of 1000° C. and when the wire is reduced to the size of about 0.005 in. (0.127 mm.) diameter, the temperature of drawing will have been reduced to a dull red heat. Fig. 6 is a micrograph of a drawn tungsten wire. All of the tungsten wires so produced, no matter what their size, are ductile cold. The increase in tensile strength is continuous, the smallest wires being the strongest.

TABLE 1.—*Tensile Strength of Tungsten*

Kind of Material	Diam. in Mils	Tensile Strength in Lb. per Sq. In.
Sintered tungsten ingot. . . . .	200 by 250	18,000
Swaged rod. . . . .	216.0	50,600
Swaged rod. . . . .	125 0	107,000
Swaged rod. . . . .	80 0	176,600
Swaged rod. . . . .	26 0	215,000
Drawn wire. . . . .	18.0	264,000
Drawn wire. . . . .	7.23	340,000
Drawn wire. . . . .	5.78	366,000
Drawn wire. . . . .	5 50	378,000
Drawn wire. . . . .	3 96	483,000
Drawn wire. . . . .	1 14	590,000

Table 1 shows the increase in tensile strength of tungsten products with the increase in mechanical working. Tungsten wire 1.14 mils diam. (a mil is 0.001 in.) shows a reduction of area at the point of fracture of about 65 per cent. Coupled with great strength, drawn tungsten wire possesses the property of ductility in the cold to a very usable degree. The wire can be coiled on mandrels having diameters but

slightly greater than that of the wire itself. It can be bent into the various shapes necessary for lamp making, and it can be handled in long lengths without danger of breaking.

Table 1 shows that the tensile strength of the 1.14-mil drawn wire is about 33 times as great as that of the sintered tungsten ingot. In ordinary metal-working processes, an increase of tensile strength of six times that of the original starting material is unusual.

#### GENERAL RELATIONSHIPS BETWEEN TUNGSTEN AND OTHER METALS

It should be noted from the preceding that tungsten possesses several properties different from those of the common ductile metals. For example, when the common ductile metals are worked cold or at elevated temperatures below their annealing temperatures, they are rendered more brittle at room temperature. Tungsten cannot be worked at room temperature when it is composed of equiaxed grains. When it is worked at elevated temperatures below its equiaxing temperature in such a manner as to deform the grains permanently, its ductility at room temperature increases.

When the ductile metals which have been made brittle by mechanical working at room temperature, or at elevated temperatures below their annealing temperatures, are annealed, ductility is restored. When tungsten which has been made ductile by mechanical working at an elevated temperature below its equiaxing temperature is heated above the equiaxing temperature, its ductility at room temperature is lost.

The ductile metals are in their most ductile condition when they are composed of small equiaxed grains. Tungsten is in its most brittle condition when it is composed of small equiaxed grains.

When the ductile metals are heated in such a manner that coarse-grained structures are produced, they become less malleable and ductile at room temperature, as a result of the coarse-grained structures. When tungsten, however, is heated in such a manner as to produce extremely coarse-grained structures, the resulting tungsten product possesses, to a slight degree, the ability to be deformed, especially by pressure at room temperature. The utility of this type of deformability in tungsten is limited. Let us suppose, for the sake of argument, that we have a single grain of tungsten, and that we proceed to work this grain mechanically at room temperature. It becomes harder and more brittle, due to the strain hardening, and our first thought would be to heat it above its equiaxing temperature, so that it would regain its malleability and ductility. Above the equiaxing temperature this strain-hardened grain would form many equiaxed grains. We would have produced in this manner a piece of tungsten metal composed of small equiaxed grains. This material would be absolutely brittle at room temperature and no further working at room temperature would be possible. This piece of tungsten could,



however, be mechanically worked at high temperatures by the processes described above.

Molybdenum has some properties similar to those of tungsten, and others similar to those of the common ductile metals. It is in a more ductile condition at room temperature when it is composed of fine grains than when composed of coarse grains. In this respect it is similar to the common ductile metals. When its grains are deformed by mechanical working at temperatures above room temperature and below its equiaxing temperature, its ductility at room temperature increases. In this respect it more nearly resembles tungsten.

Tantalum<sup>35</sup> resembles the common ductile metals in all of its working properties. Tantalum must be very pure and especially free from hydrogen or nitrogen to be in a ductile condition. When it is melted in a vacuum, it is found to be one of the most malleable and ductile of metals. Fig. 7 is a micrograph of a cold-drawn tantalum wire which was mechanically worked at room temperature, without any annealing, from a fused globule of tantalum to a wire 0.007 in. (0.177 mm.) in diameter. This wire has not even yet reached its maximum limit of cold working. Fig. 8 shows a piece of the same wire which was heated in a vacuum to 1600° C. for 5 min. It will be noted that recrystallization has taken place. This annealed wire is much more ductile than the cold-worked wire, thus showing that in these respects tantalum is similar to the common ductile metals. Fig. 9 is a micrograph of a piece of the same tantalum wire heated for 1 min., in an atmosphere of hydrogen, to 1600° C. Recrystallization has taken place, but this wire has absorbed enough hydrogen to make it extremely brittle and fragile at room temperature. The properties of tungsten wire are the same whether heated in a vacuum or in hydrogen.

Cast zinc or worked zinc made coarse grained by a high-temperature annealing may be brittle at room temperature and workable at 150° C., and after this working the ductility at room temperature will be greater than before working. It is now known that this increase in ductility of zinc is due to a substitution of a fine-grained structure for the coarse-grained one. It is known that 150° C. is above the annealing temperature of worked zinc and consequently in the hot-working region. The refinement of the grain in zinc by working at 100° to 150° C. is analogous to the breaking down of the coarse grains in a steel ingot by working above a red heat.<sup>36</sup> No amount of work on tungsten above its annealing temperature will make it ductile at room temperature.

Platinum may be produced in a coherent malleable and ductile form by the Wollaston process<sup>37</sup> without resorting to the fusion operation. There are several points of difference between the Wollaston method for producing coherent ductile platinum without fusion and the method of producing coherent ductile tungsten without fusion. The platinum has

only to be made coherent and it is inherently ductile. Tungsten has to be made coherent and then ductilized by the working process. The platinum cake immediately before working contains about 40 per cent. voids and these are welded by the working. The voids must be eliminated in tungsten before the working begins, else the material will not be workable.

#### EFFECT OF THORIA ON THE STRUCTURE OF SINTERED TUNGSTEN INGOTS

The principal need for thoria or other non-metallic substances in tungsten which is to be used for lamp filaments is to control the direction, and, to a certain extent, the degree of grain growth in the filament, in such a manner as to render them both rugged (not easily broken by rough handling of the lamps) and free from off-setting.\* With the old type of pressed tungsten filament, the purity of the tungsten did not have to be considered except for the functioning of the tungsten wire as a lamp filament. By the present method of manufacture of lamp filaments, however, the sintered tungsten ingots must be capable of being mechanically worked hot. Therefore the addition of thoria or other non-metallic substance must be made judiciously and the heat treatment of the tungsten ingot must also be varied to suit the thoria content. Tungsten containing more than 2 per cent. thoria is difficult to work into small wires.

The effect of the thoria is to increase the resistance to grain growth at high temperatures. Other non-metallic substances will offer resistance to grain growth in proportion to the volume, arrangement, and size of globules in the metal. It is difficult, however, to retain the more volatile oxides in the ingot. Even silica vaporizes almost completely during the sintering operation. Alumina volatilizes more slowly and thoria very little.

Plate 1 shows the variation in grain size of tungsten ingot with variation on the quantity of thoria, the temperature being maintained constant at about 3200° C. and the time constant—12 min. It would seem as if the resistance to grain growth had decreased in the region between 2.5 and 4 per cent. thoria. This apparent decrease in resistance to grain growth is readily explained by the germinative temperature laws.<sup>17-26</sup> The increase in resistance to grain growth is continuous with increase in the amount of thoria, according to the dotted portions of the curve shown in Plate 1. The germinative conditions have not produced exaggerated growth of grain up to about 1.5 per cent. thoria,

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\* Off-setting is the transverse displacement of a section of the filament wire. The displacement occurs at places where a grain boundary occupies the whole cross-section of the wire.

nor above about 4.5 per cent. thoria, so that the normal curve representing equilibrium grain size for the given time and temperature conditions would be independent of the exaggerated grain growth portion. These changes in grain size due to variation in the quantity of thoria can be seen plainly in the micrographs. Figs. 10, 11, 12, 13, and 14 are micrographs of the tungsten ingots containing respectively 1, 2, 3, 4, and 5 per cent. thoria. The ingots were all sintered at 3200° C. for 12 min. Figs. 15, 16, 17, 18 and 19 are micrographs of the same samples containing 1, 2, 3, 4, and 5 per cent. thoria respectively, at a higher magnification.

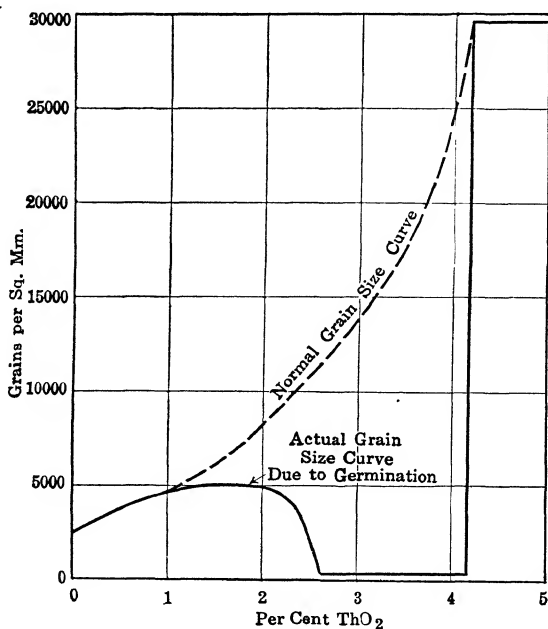


PLATE 1.—VARIATION IN GRAIN SIZE OF TUNGSTEN INGOTS WITH VARIATION IN THORIA. HEATED 12 MIN. TO MEAN TEMPERATURE OF 3200° C.

The micrographs at the higher magnification show very plainly not only the quantity of thoria, but its distribution. The thoria is present in small spherical globules in all of the samples. The number of globules is much greater in Fig. 19 than in Fig. 15. The size of the globules, however, does not vary in a marked degree.

The explanation for these coarse-grained structures with increase in resistance to grain growth is as follows: Let us take, for example, the sample containing 3 per cent. thoria. When it was heated to a mean temperature of 3200° C. by the passage of an electric current in an atmosphere of hydrogen, the axis of the comparatively long tungsten ingot would be its hottest portion. A temperature gradient would obtain from the axis to the surface of the ingot. With 1 per cent.

thoria present, even the surface of the ingot, when heated to a mean temperature of  $3200^{\circ}\text{C}$ . would permit the free growth of adjacent grains. With 3 per cent. thoria, however, the resistance to grain growth will have been sufficiently increased so that the portion of the tungsten ingot near the surface will be below the temperature of free grain growth. The portions nearer the axis, being hotter, will permit grain growth to take place. There will be a sharp boundary line between that portion of the ingot in which grain growth could take place and that portion in which growth was not possible (or at least could take place but slowly) under the existing conditions. The grains in the growth range will be larger than those in the inert range. At the boundary between the growth and inert grains, the former, because of their larger size, will be able to absorb the latter, which cannot coalesce with one another because they are (1) too nearly the same size, and (2) too cold. Adjacent grains in the growth range away from the boundary between the growth and inert grains, cannot coalesce freely with one another because they are too nearly the same size. Their size, however, is considerably greater than that of the grains in the inert region. The grains at the boundary between the inert and growth regions become germinant grains. They increase their size by absorption of the inert grains until they are much larger than the hotter grains in the growth region, which they then absorb. The resulting structure will be an extremely coarse-grained one, with a general tendency for the grains to be radial, much as if the ingot had solidified in a mold from the molten condition.

In the sample containing 2.5 per cent. thoria, germinant grains were formed first at a lower temperature, because the resistance to grain growth would be less with 2.5 per cent. than with 3 per cent. thoria. With the same average temperature, the germinant grains in the 2.5 per cent. thoria sample would first form nearer the surface of the sample. This is exactly what was observed. With the sample containing 4 per cent. thoria, the germinant grains would first form in a hotter portion of the ingot. It was observed that the germinant grains did form initially in the 4 per cent. thoria ingot practically at its axis. This indicates that with the 4 per cent. thoria ingot at a mean temperature of  $3200^{\circ}\text{C}$ . the region of free grain growth occurs only in the hottest portion of the ingot and all other parts of the ingot are in the inert range. With 5 per cent. thoria, no part of the ingot is sufficiently hot with a mean temperature of  $3200^{\circ}\text{C}$ . for free grain growth to take place in a time period of 12 min., so the whole cross-section of the ingot is in the inert range. The samples containing 1 and 2 per cent. thoria, at a mean temperature of  $3200^{\circ}\text{C}$ . were sufficiently hot for the whole cross-sections to be above their germinative temperatures, that is, to be entirely within the growth range. It will be noted that the 2 per cent. thoria sample is finer grained than the 1 per cent. sample and the sample containing 5

per cent. thoria is the finest grained of them all. It will be noted also in these samples that where grain growth has taken place the thoria globules have not apparently changed in position. For example, in Fig. 17 a portion of one grain only is shown. The thoria globules, however, are distributed within this grain in a manner very similar to the distribution of the thoria globules in Fig. 19, which consists of a fine-grained structure with the thoria globules largely situated at the boundaries of the small grains.

Some calculations indicate that the difference in temperature between the surface of the tungsten ingot  $\frac{1}{4}$  in. (6.35 mm.) square and its axis is about  $150^{\circ}$  C. when a mean temperature of  $3200^{\circ}$  C. is maintained by the passage of electric current through the ingot.

By heating these samples for various time periods, the history of grain growth can be studied. In the growth range, an apparent equilibrium grain size is reached within a few minutes at a high temperature, say  $3200^{\circ}$  C. At lower temperatures around  $2600^{\circ}$  and  $2700^{\circ}$  C. a longer time is needed in order to produce an apparent equilibrium grain size. Under germinative conditions, if the germinative temperature is high, the velocity of grain growth is rapid, and if the germinative temperature is low, the velocity of grain growth is slow. At a temperature of  $2600^{\circ}$  C. the rate of grain growth was only about one-twentieth of the rate at  $2300^{\circ}$  C.

The sintering together of the individual particles of tungsten, or their welding, is an entirely distinct operation from their coalescence. At a temperature of about  $2400^{\circ}$  or  $2500^{\circ}$  C., sintering takes place and is complete within a few minutes. Most of the voids have been closed and most of the shrinkage of the ingot takes place during the sintering. With increase in temperature, these small grains formed by the sintering operation commence their growth. If no germinative temperature condition is encountered, the grains will increase in size with increase in temperature, and up to a certain point with increase in time of exposure. Germinative conditions, however, may change the order of grain size enormously.

When the germinative temperature is maintained at a point about midway between the axis and the surface of an ingot, the large germinant grains grow both toward the surface, absorbing the inert grains, and toward the axis, absorbing the grains which are in approximate equilibrium with each other. At the boundary lines between two large germinant grains and a fine-grained region, there will always be, if the fine-grained region is considered as a sea and the coarse-grained region as land, a bay at the intersection of the two large grains with the fine-grained region. This indicates that the two large grains may be competing for the small grains at this point. The forces of attack will act in

different directions and a sort of neutral zone in which grain growth is less rapid than normal will obtain. Fig. 20 shows this phenomenon.

Tungsten ingots made up of an aggregate of large and small grains are not easily worked. They are apt to crack at low temperatures along the grain boundaries, between the large grains or at boundaries between large- and small-grained areas.

#### EFFECT OF CHANGE OF TEMPERATURE ON THE STRUCTURE OF TUNGSTEN INGOTS

Since a temperature of  $3200^{\circ}\text{C}$ . produces the germinative conditions at the axis of a tungsten ingot containing 4 per cent. thoria, a fine-grained structure would be produced by heating to temperatures lower than about  $3150^{\circ}\text{C}$ . The germinative temperature of a tungsten ingot containing 5 per cent. thoria, or more, is theoretically above the melting point of tungsten, so it is non-existent.

A tungsten ingot containing 2.5 per cent. thoria has its germinative temperature near the surface of the ingot when a mean temperature of  $3200^{\circ}\text{C}$ . is maintained. Therefore, if the temperature were lowered to about  $3100^{\circ}\text{C}$ ., the germinative temperature would occur near the axis in this sample. If, however, the temperature could be increased to  $3300^{\circ}\text{C}$ . before germination had begun during heating, a fine-grained structure would result, which would correspond to the normal equilibrium grain size in the growth range for the given time and temperature conditions.

With the sample containing 1.5 per cent. thoria, a mean temperature of  $3200^{\circ}\text{C}$ . was sufficient to make every part of the cross-section of the ingot above its germinative temperature. If the temperature could have been lowered to, say,  $2900^{\circ}\text{C}$ ., germinative conditions would have obtained in this sample with the resultant production of a coarse-grained structure. The germinative temperature of tungsten ingots containing 0.75 per cent. thoria is about  $2550^{\circ}$  to  $2650^{\circ}\text{C}$ . This germinative temperature is so low for tungsten that the individual particles of the tungsten powder must be very small or the formation of large grains at the germinative temperature will be defeated.

Some tungsten powder was prepared consisting of grains or particles very much smaller than the size usually employed. Experiments were made with tungsten ingots pressed from this very fine tungsten powder. It was found that the germinative temperature occurred at about  $2600^{\circ}\text{C}$ . The time necessary to produce a marked coarsening of the grain at the germinative temperature was very much longer in this material than with material whose germinative temperature was in the neighborhood of  $3200^{\circ}\text{C}$ .

Fig. 21 represents the fracture of one of these tungsten ingots heated for 20 min. at a temperature near  $2600^{\circ}\text{C}$ . Fig. 22 is the fracture of an

ingot treated at a temperature of about 2600° C. for 30 min. In both of these photographs, the coarse-grained areas can be seen in the region midway between the axis and the surface of the sample. The axis itself is fine-grained and the surface is fine-grained.

Fig. 23 is a fracture of one of these tungsten ingots which was heated quickly to a temperature of 3200° C. and held for 30 min. The structure is fine-grained and uniform.

Fig. 24 is a micrograph of a tungsten ingot held at 2600° C. for 20 min.

Fig. 25 shows the cross-section of an ingot heated to about 2600° C. for 30 min. The fine-grained portions near the surface in both these samples is finer grained than that near the axis.

Fig. 26 is a longitudinal section through section A-A of the same ingot as Fig. 25.

Fig. 27 shows how the germinative temperature conditions produce coarse-grained structures at the ends of the ingots which are held in the electrodes. There is a temperature gradient at the ends of the ingots, because the electrodes are either directly or indirectly water cooled so that a change of temperature of at least 2000° is maintained between the portion of the ingot held in the electrode clamp and the hottest portion. Fig. 27 shows where the germinative temperature existed. To the left of the coarse-grained region may be seen the inert region, and to the right the gradual change of the coarse-grained area to a normal fine-grained structure. It is needless to say that the sintering temperature of this ingot was far above its germinative temperature.

Fig. 28 shows the structure of a tungsten ingot, heated quickly past the germinative temperature and held there for 30 min. Any intermediate structure between the coarse-grained structure formed at the germinative temperature and the normal fine-grained structure formed by heating quickly through the germinative temperature range can be produced by varying the time in which the ingot remains in the germinative temperature range during heating—that is, by varying the rate of heating.

Figs. 29 and 29A are micrographs of a tungsten ingot which was held for 20 min. at 2600° C., and then heated quickly to 3200° C. and held there for 10 min. The grain size of this sample is very much coarser than the normal grain size formed by rapid heating through the germinative temperature range and much finer than the grain size which was formed by a long sojourn in the germinative temperature range.

The smallest grain size that can be produced in an ingot will depend upon the size of the tungsten particles composing the tungsten powder. At the time these particles weld themselves together (sinter), the smallest grain size which it is possible to obtain from this tungsten powder will result. A tungsten ingot in this condition, however, finds very

little use in the industries. It is too fine-grained to be easily amenable to the tungsten working process, especially when it is desired to secure a great amount of permanent deformation. For example, a tungsten ingot treated in this manner can be swaged and drawn only with the greatest difficulty from an ingot  $\frac{1}{4}$  in. square to a small drawn wire. Such a metal is so hard that the diamond die wear may be 50 times as great during drawing as that of tungsten ingots composed of larger grains.

By heating the tungsten ingot just above the germinative temperature, the smallest grain size in the growth range is produced. It is only in the growth range that the tungsten ingots lend themselves readily

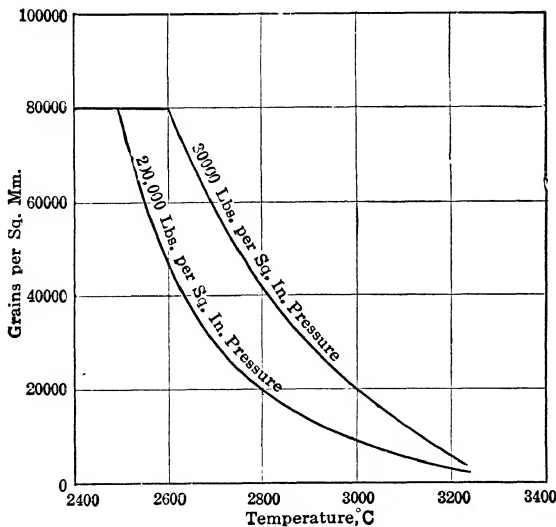


PLATE 2.—GRAIN-SIZE TEMPERATURE CURVES OF TUNGSTEN BRIQUETS. FIGURES REFER TO INITIAL PRESSURE USED IN BRIQUETTING. TIME OF HEATING, 12 MIN.

to the mechanical working process. As the temperature increases, the grain size of the tungsten ingots increases up to the melting point of the tungsten.

The relatively coarse tungsten powder ordinarily employed in the manufacture of tungsten ingots is sufficiently coarse grained to mask the germinative temperature phenomena in the 0.75 per cent. thoriated ingots during sintering. In these ingots, the grain size starts at a minimum at about 2600° C. and gradually increases up to the melting point. A typical set of results is shown in Plate 2. The curves show the variation in grain size in tungsten ingots due to variation in the pressure to which the tungsten powder is subjected, and to variation in the temperature to which the tungsten is heated. We may call a pressure of 30,000 lb. to the square inch a normal pressure. The grain size of an ingot



sintered for 12 min. at 2500° C. will be about 80,000 grains per square millimeter. At 2600° C., the grain size is the same. This indicates that below 2600° C. tungsten containing 0.75 per cent. thoria is in its inert range. The grain size will become progressively larger as the temperature is increased above 2600° C., so that at 3200° C. a grain size of 5000 per square millimeter or less will usually result with tungsten metal containing 0.75 per cent. thoria.

By increasing the pressure used in making the compressed tungsten slug to 200,000 lb. per square inch, the grain size becomes larger for any given temperature of treatment, as shown in Plate 2. The use of 200,000 lb. pressure in practice, however, is not only an extremely difficult task to perform but it is absolutely unnecessary for the accomplishment of any given purpose; for example, any result produced with 200,000 lb. per square inch pressure can be duplicated at a higher temperature with a smaller pressure.

The tungsten powder is partly amorphous and partly crystalline. The crystalline particles are probably strain-hardened by the pressure, but these strains play little part in the grain growth. They are removed before sintering takes place. The reason that high pressure tends to make larger grains is that the tungsten particles are pressed into more intimate contact, thus facilitating grain growth.

By comparing Plates 1 and 2, it will be observed that the increase in resistance to grain growth of about 4 per cent. thoria is equivalent to the decrease in resistance to grain growth due to lowering the temperature about 300° C.

#### CHANGES IN GRAIN SIZE DUE TO INCOMPLETE FUSION OF TUNGSTEN INGOTS

Tungsten ingots containing about 0.75 per cent. thoria remain comparatively fine grained even up to the melting point. Fig. 30 is a micrograph of one of these ingots in which the central portion has fused and resolidified and the outer portion has remained entirely in the solid state. Fig. 30A represents the same structure at a higher magnification. We have been taught that when fused metal at the time of solidification is in contact with crystalline metal of the same composition, this latter will act as a nucleus about which the molten metal will crystallize and assume the same orientation as the crystalline nucleus. In Fig. 30, when the tungsten was molten, it was in contact with hundreds of the small grains composing the unfused portion of the ingot. When solidification began, why did not each one of these small differently oriented grains act as a nucleus for the crystallization of the molten tungsten? They probably did. If the crystallization of the fused tungsten began from many points with different orientation, why was the resulting struc-

ture so coarse-grained as that shown in Fig. 30? The answer in my opinion is as follows: When a molten metal is in the process of solidification, it is in its most susceptible condition for grain growth. In other words, the possibility of one grain absorbing adjacent grains in an almost inconceivably short time is greater at the melting point of a metal than at any other temperature. Let us suppose that the crystallization of this fused tungsten began at the contact between the fused and the unfused portion. This must necessarily have been the case. The small grains would grow axially and if no grain growth could take place during the solidification, each of these grains would form a wedge, the small end being at the axis of the ingot. This has not taken place. Some of these grains having advantages over their neighbors, have acquired crystalline material from the molten metal faster than others. These more vigorous grains have shot out arms perpendicular to their axes of growth and have absorbed not only the molten metal which should have gone to some of the other nuclei, but also some of the crystalline material which had solidified on other nuclei. In this manner only a few of the more vigorous grains have survived.

Fig. 31 shows that if the original structure of the ingot is coarse-grained instead of fine-grained, the fused central portion will resolidify strictly in accordance with the laws of the initial nuclei. For example, it will be seen that every grain in the unfused portion has its continuation in the portion which fused and subsequently resolidified. There is no reason to suppose that at the beginning of solidification of Fig. 30, crystallization did not start from each and every small grain which was in contact with the fused metal. This leads us to the conclusion that, in cast metals, the grain size does not depend entirely on the number of nuclei from which crystallization begins, but that grain growth takes place at a remarkably fast rate during solidification.

### TUNGSTEN AND CARBON

Tungsten in the presence of carbonaceous gases at high temperatures, or in contact with solid carbon at high temperatures, forms tungsten carbide, generally  $W_2C$ . Tungsten containing even small amounts of tungsten carbide at the boundaries of the grains is extremely brittle and fragile both at room temperature and at high temperatures. It has not been possible to mechanically work tungsten ingots containing tungsten carbide as a net work. Fig. 32 is a micrograph of a tungsten ingot made by adding 0.9 per cent. carbon in the form of lamp black to the tungsten powder before pressing. The tungsten carbide is the lighter constituent surrounding the grains of pure tungsten. This material is brittle, both hot and cold.

When too much carbon is added, the tungsten ingot, especially if

thoria is present in the tungsten powder, will burst during the sintering operation. Fig. 33 is a photograph showing one of the bursted ingots containing carbon and a normal tungsten ingot without carbon.

If a piece of tungsten metal is heated to a temperature of  $1800^{\circ}\text{C}.$ , in contact with solid carbon or in the presence of carbonaceous gases, tungsten carbide forms readily at the surface of the sample. Carbon containing gases will act more quickly and at lower temperatures than solid carbon. Hydrocarbons may form tungsten carbide in contact with metallic tungsten at temperatures as low as  $1100^{\circ}\text{C}.$  Fig. 34 shows a piece of tungsten that was heated for 1 hr. in an electric furnace in an atmosphere of CO. The lighter portion is the tungsten carbide which forms on the surface. The darker portion is pure tungsten. Fig. 35 shows a piece of tungsten metal heated for  $1\frac{1}{4}$  hr. at a temperature near  $1800^{\circ}\text{C}.$ , the tungsten being placed on a platform of carbon while an atmosphere of hydrogen was maintained in the furnace. The lighter portion is tungsten carbide. The depth of carbonization increases with increase in time. In  $1\frac{1}{4}$  hr. the depth of carbonization was 0.21 mm. and in 3 hr. 0.30 mm. at  $1800^{\circ}\text{C}.$

It has been found possible to work tungsten mechanically containing a considerable amount of tungsten carbide when the carbide was made separately, powdered, and added to the tungsten powder before pressing. Tungsten ingots containing tungsten carbide made in this manner may be mechanically worked when the carbon content is far in excess of that necessary to make the tungsten brittle ordinarily. When added in this way, the tungsten carbide does not surround the tungsten grains.

#### GENERAL CONSIDERATIONS REGARDING THE WORKING PROPERTIES OF TUNGSTEN

The sintered tungsten ingots are usually comparatively fine grained, say from 1500 to 5000 grains per square millimeter. It is considered desirable at present to keep the grain size of the ingots comparatively small because the inherent resistance to grain growth will usually give better life properties to the tungsten wire when used for lamp filaments. It is easily possible, however, to get the grains too fine for proper mechanical working. The more thoria or other non-metallic materials present, the more does fineness of grain militate against the mechanical working. With 0.75 per cent. thoria, which is the amount ordinarily used, if the grain size in the ingot is more than about 7000 per square millimeter, the metal will be extremely hard to work, especially in the smaller sizes. It is usually advisable to keep the grain size below 6000 per square millimeter. Tungsten ingots have been worked containing as high as 30,000 grains per square millimeter, but the metal is so hard that it is impractical to work it as a commercial process.

With very coarse-grained structures—for example, with ingots containing grains each of which measures several square millimeters in area—the first stages of the mechanical working process are very apt to break the metal. If one of these coarse-grained ingots passes the first eight or ten pairs of swaging dies, the rest of the working process is comparatively easy—in fact, easier than the working of the tungsten rods made from finer-grained ingots.

If it is desired to produce ductility in tungsten with a comparatively small amount of working, the following processes may be resorted to:

1. The working should be carried on at as low a temperature as possible.

2. The grain size of the ingot should be as small as will permit mechanical working without causing excessive trouble.

Either method, or both of the methods carried out simultaneously, will produce the desired effect, but the resulting product, which has been made ductile with a minimum amount of mechanical working, is not a suitable material for further mechanical working. It will be so hard that it will wear the dies unduly, and, furthermore, it will break and split easily during the working. What is more apt to be desired than the production of ductility in tungsten with a comparatively small amount of mechanical working is the possibility of working the tungsten a great deal before it becomes too hard and intractable for further working. This end is accomplished in several ways:

1. The grain size of the sintered tungsten ingot should be less than about 6000 grains per square millimeter.

2. The working temperature at the beginning of swaging should be very high, say 1700° C.

3. The initial swaging temperature may be considerably lower and the swaged tungsten rod can be subjected at intervals to a heating process which will cause the distorted grains formed by working to change into equiaxed grains, thereby forming a structure usually somewhat coarser-grained than that of the ingot. The equiaxed tungsten rod is then capable of a greater amount of mechanical working at the proper temperature than it would have been had it not been equiaxed.

In order to produce ductile tungsten by this process, it is necessary that it be subjected to considerable mechanical work after the last equiaxing treatment. Aside from the difference in the shapes of the grains in worked tungsten which has subsequently been equiaxed, and sintered tungsten, the two are similar, that is, the ductility at room temperature which has been gained by mechanical working below the equiaxing temperature of tungsten is lost by equiaxing; it can only be regained by further mechanical working at the temperatures indicated.

If it is desired to produce a piece of tungsten which is ductile at a particular size, this may be accomplished in the following ways:

1. By starting with a coarse-grained material of such a size that it will take a considerable amount of mechanical working to reduce it to the desired size.

2. By starting with a fine-grained material of such a size that a moderate amount of mechanical working, say an elongation of 25 times its original length, will reduce it to the desired size.

3. By starting with smaller sizes than would be used with (1) and (2) and performing the mechanical working operations at temperatures as low as possible.

The reasons for the above generalities have been largely ascertained and will be considered later.

From the time the tungsten wire reaches a diameter of 0.030 in. (0.76 mm.) or less, it is handled on reels. It would not be at all convenient, at any stage in the fine-wire manufacture, to heat it to such a temperature that the fibrous structures to which tungsten owes its ductility are destroyed. Such an operation would make the wire brittle and it could not be handled on reels in lengths of a mile or more at room temperature (as is done now) but would have to be handled in comparatively short lengths. This would slow up production to such an extent that it could not be tolerated.

Let us suppose that we have two tungsten ingots made from the same tungsten powder but that one of them has been sintered at the germinative temperature and, therefore, is coarse grained. Let us suppose that the other ingot has been sintered at a temperature above the germinative temperature and consequently is fine grained. Ingots of this nature have been made, in which the grain size was 50,000 times as great in the coarse- as in the fine-grained sample. These two ingots can be worked and treated in such a manner that when small wires are produced from them, they will be practically identical in structure. Both ingots are first swaged until their length has been about doubled or has reached some other suitable length; then these swaged rods are heated in an atmosphere of hydrogen by the passage of an electric current to a temperature above that of equiaxing. The result in an individual case was as follows:

Before treatment, the grain size ratio in the two ingots was 50,000 to 1; after swaging to 100 per cent. elongation and reheating to above the equiaxing temperature, the grain size ratio was 4:1. Fig. 36 shows a longitudinal section of the fine-grained ingot after swaging and reheating, and Fig. 37 a longitudinal section of the coarse-grained ingot after swaging and reheating. After an additional swaging and reheating, the grain size of the two rods will be practically identical. This process can be used to equalize the structures of the tungsten rods when desirable. Fig. 38 is a typical example of a longitudinal section of a swaged tungsten

rod. Fig. 39 is a micrograph of a similar tungsten rod after heating for 3 min. to a temperature of  $3100^{\circ}\text{C}$ .

Tungsten rods which have been worked and recrystallized are stronger than sintered rods. In the sintered rods, the grains are exactly equiaxed, but in the recrystallized rods the grains are longer in a longitudinal than in a transverse direction.

### *Equiaxing Temperature of Worked Tungsten*

The equiaxing temperatures of worked tungsten have been determined with an exposure of 5 min. The equiaxing temperature of a swaged tungsten rod which has been reduced in area by working about 24 per cent. is  $2200^{\circ}\text{C}$ . The equiaxing temperatures are gradually lowered both as the degree of deformation increases and as the temperatures of working decrease. Both of these factors serve to give tungsten a long equiaxing temperature range. When the swaging process has been carried on so as to reduce the area by working about 90 per cent., the equiaxing temperature is  $1800^{\circ}\text{C}$ . for a period of 5 min. With the very small tungsten wires which have been reduced in area 99.99 per cent. or more, and which have been finished at a comparatively low temperature, say a dull red heat, the equiaxing temperature in a period of 5 min. will be in the neighborhood of  $1350^{\circ}\text{C}$ . The equiaxing temperature also decreases as the time of exposure increases.

### *Germinative Temperature Phenomena in Worked Tungsten*

When most metals have their grains permanently deformed, a slight amount, say corresponding to a reduction of area of 1 per cent. or more, but usually not more than 20 or 30 per cent., germinative temperature phenomena are apt to render them coarse grained during a normal annealing process. With ordinary muffle heating, there is apt to be no temperature gradient, or at most a very slight one, and a moderate deformation will cause within the metal a strain gradient which will cause germinative grains to form at suitable temperatures with the consequent production of coarse-grained structures.

When tungsten is deformed a moderate amount, say a few per cent. reduction in area, it should form these coarse grains at its germinative temperature, and it probably would if it were heated in a muffle to a temperature of about  $2300^{\circ}\text{C}$  or  $2400^{\circ}\text{C}$ . When such a piece of tungsten, however, is heated by the passage of an electric current, the strain gradient has its maximum strain near the surface of the sample (at the point of application of the deformational forces) and the point of minimum strain is at the axis of the tungsten rod. When this rod is heated by passing an electric current through it, its axis is heated to a higher temperature than its surface. The temperature gradient, therefore,

serves to counterbalance the strain gradient in such a manner as to defeat the formation of coarse grains.

As the degree of deformation increases, however, the strain gradient from surface to axis of the tungsten rod becomes practically nil. The temperature gradient during heating or during a sojourn at a certain high temperature will still obtain. Germinative temperature phenomena are then apparent in worked tungsten. When a tungsten rod has been swaged from 0.250 in. (6.35 mm.) to 0.080 in. (2.03 mm.), coarse grains can be produced by maintaining the 0.080-in. rod at a temperature just above that of equiaxing for an extended period of time. Coarse grains may also be produced in these 0.080-in. swaged rods by heating gradually with electric current to a very high temperature. Fig. 40 is a micrograph of a sample of 0.080-in. swaged tungsten rod heated to a temperature of 3100° C., the time of heating being about 10 sec. Fig. 41 shows a piece of the same 0.080-in. swaged tungsten rod which was heated as quickly as possible to 3100° C. It will be noted that the sample that was heated quickly is comparatively fine grained and the sample that was heated more slowly is extremely coarse grained. The sample shown in Fig. 41 can be maintained at a temperature of 3100° C. for hours without changing its grain size noticeably. This shows that a variation in the change of rate of heating of 10 sec. may have more influence on the resulting grain size than an exposure of many hours, or even many days, at the highest temperature reached. All rates of heating requiring more than 10 sec. to reach 3100° C. will produce coarse-grained structures in these 0.080-in. rods. The heating of an 0.080-in. swaged tungsten rod to a temperature of 2800° C. in a period of 10 sec. or more will result in a coarse-grained structure.

It should be noted in this respect that the presence of thoria in swaged tungsten operates against the formation of these large grains. It should also be noted that all of the heating is done by passing electric current through the metal itself in an atmosphere of hydrogen, thus causing a temperature gradient to exist in the heated sample. These examples of rapid grain growth are with tungsten ingots containing no thoria.

At a temperature of 2750° C., the rate of heating must be 11 sec. or more in order to produce the coarse-grained structure. At a temperature of 2700° C., if the time of reaching that temperature is less than 7½ min., the structure will be fine grained, whereas if the time is more than 7½ min., the structure will be coarse grained. At a temperature of 2600° C., the time of heating to that temperature must be more than 20 min. in order to produce a coarse-grained structure. In these remarks, a coarse-grained structure indicates the very coarse-grained structure produced under exaggerated grain-growth conditions. These time periods will also change with the particular material used.

If the 0.080-in. tungsten wire is held at a mean temperature slightly

above the equiaxing temperature for an extended time, large grains will form according to the regular germinative temperature law. Fig. 42 will illustrate this grain growth at constant mean temperature, and a constant temperature gradient. This is a micrograph of an 0.080-in. swaged tungsten rod which has been reheated to a comparatively high temperature. The micrograph shows that portion which was held in the electrode clip and also the portion near the clip. It will be seen that on the left of the micrograph the grains are deformed. This was obviously the coldest portion. Temperature gradients in two directions would obtain near the electrode clip. There would be an axial temperature gradient and a radial one. The portion at the extreme right of the micrograph was above the germinative temperature and this portion exhibits a rather uniform grain size throughout. At all portions, however, between the unrecrystallized portion and the extreme right of the micrograph, the germinative temperature has existed in some part of the rod. In the hotter zone, the germinative temperature has been near the surface, and in the cooler regions, near the axis of the rod. The germinative temperature areas can be seen readily in this micrograph, although sufficient time was not given for the germinative temperature regions to encroach upon the smaller grains of either the hotter or colder portions of the sample. This figure, therefore, represents the first stages of grain growth in the germinative temperature region. Fig. 43 is a micrograph of a similar sample after the germinative grains have reached a larger comparative size. Fig. 44 is a micrograph of the same sample as Fig. 43 in the hotter portion which was above the germinative temperature. It will be noted that the colder germinative temperature portion has produced larger grains than the hotter portion which was above the germinative temperature.

When an 0.080-in. swaged tungsten rod is held at its germinative temperature, it takes considerable time for the coarse grains to form on the entire cross-section of the rod. As the size of the tungsten wire becomes smaller, sometimes a few seconds at the germinative temperature is sufficient to cause the formation of coarse-grained structures. The presence of thorium also impedes the formation of large grains in these small tungsten wires.

Table 2 shows the minimum time required to change tungsten wires originally possessing fibrous structures to coarse-grained structures, and also the variation in the diameter of wire and in the temperature to which it is heated. The tungsten wires used in these experiments did not contain thorium. The thoriated wires sometimes take as long as 1000 or 1500 hr. to change into the coarse-grained structures unless the temperature be maintained at the germinative temperature. In this event, a few hours only are required to form the coarse-grained structure. It should be noted from Table 2 that tungsten wires 0.004 in. in diameter



form coarse-grained structures easily, whereas tungsten wires 0.005 in. in diameter or greater seem reluctant to form the coarse grains when the temperature is maintained constant either at or above the germinative temperature. The coarse grains form more quickly at the germinative temperature than they do at higher temperatures, but there is a marked difference in time required to form the coarse-grained structures at the germinative temperature between the tungsten wires less than 0.005 in. (0.127 mm.) diameter, and more than 0.005 in. diameter.

TABLE 2

Diam. of Wire in Mils	Temperature in °C.	Minimum Time Required to Change to Coarse-grained Structure	
		Minutes	Seconds
3	3,100	3	0
3	2,900	2	45
3	2,800	2	0
3	2,700	0	45
3	2,650	0	30
3	2,600	0	7
3	2,550	0	20
3	2,500	0	20
4	3,100	more than 2	30
4	2,900	3	0
4	2,800	3	0
4	2,700	2	45
4	2,650	2	45
4	2,600	2	45
4	2,550	1	45
4	2,500	1	0
5	2,900	13	0
5	2,800	more than 30	0
5	2,700	more than 30	0
5	2,650	more than 28	0
5	2,600	more than 40	0
5	2,550	more than 60	0
5	2,500	more than 40	0
6	2,900	more than 30	0

In the smaller tungsten wires, say below 0.005 in. diameter, containing no thorium, it will be noted that coarse-grained structures are formed in comparatively short time periods when the temperature is raised above the germinative temperature very quickly and maintained above the germinative temperature. The coarse grains form more readily, however, in these wires at the germinative temperature than at higher temperatures. Furthermore, a slow heating through the germinative temperature range will produce coarse-grained structures very much more readily than a rapid heating past the germinative temperature range

followed by a sojourn at the higher temperature. If the diameter of the wire is more than about 0.005 in., the easiest way to form the coarse-grained structures is by slow heating through the germinative temperature range followed by a sojourn at a higher temperature. It can be seen from Table 2 that it may take as long as an hour to form coarse-grained structures with wires 0.005 in. or greater in diameter either when held at the germinative temperature or above it. Coarse-grained structures in these samples, however, can be produced in a few seconds by heating the wire slowly through the germinative temperature range.

Fig. 45 is a typical example of the fine-grained structures produced in these experiments and Fig. 46 is a typical example of the coarse-grained structures.

The formation of coarse-grained structures during a gradual heating through the germinative temperature range needs further consideration here. The effect of heating so quickly that germination does not have time to assert itself in the germinative temperature range has already been considered. In the pressed tungsten slug, for example, a heating period of 6 min. can be used to attain the highest temperature without any exaggerated grain growth. Some of the swaged tungsten rods, however, and also the small drawn wire, must be heated through the germinative temperature range in less than 10 sec. to defeat the formation of the coarse-grained structures. The germinative temperature will be lower as the time of heating is increased. For example, if a tungsten wire is heated by electric current to a temperature just above that of equiaxing and held there for an indefinite time, coarse grains will be formed for reasons discussed above. If, however, the tungsten wire is held at a temperature several hundred degrees above the equiaxing temperature for a short time, a coarse-grained structure may be produced in a shorter time. If the tungsten rod is heated to a temperature far above that of equiaxing, fine grains will first form. They may change to large grains due to normal grain growth, but not by germination; or they may remain as small grains.

The equiaxing temperature is a function of the time, and consequently the germinative temperature which depends to a certain extent upon the equiaxing temperature will also be a function of the time. If we could heat a piece of cold-worked copper to 1000° C. and cool it again to room temperature, the whole cycle taking but a millionth of a second, we would find that the structure of the wire had not been materially changed nor would the properties at room temperature be changed. In other words, cold-worked copper which will recrystallize or equiax at a temperature of 250° C. if sufficient time be given, will not equiax or recrystallize in a millionth of a second at a temperature 750° higher. Experiments of this nature have been conducted on tungsten wire. The wire can be flashed to a temperature near its melting point

and quickly cooled to room temperature without apparent change in its structure or properties. Recrystallization of deformed grains involves a certain time period. If the temperature is comparatively low, the time is long, and *vice versa*.

When a fibrous tungsten wire is heated at a rapid rate by means of electric current, but not so rapidly that the germinative temperature laws are masked, germinant grains will form at some temperature depending upon the rate of heating. These germinant grains should be able to absorb both hotter and colder smaller grains faster than these smaller grains can coalesce with one another. When this condition obtains, coarse-grained structures will result. The gradual rise in temperature favors the absorption of the smaller grains by the larger germinant grains. The grain growth becomes a race between the germinant grains and the smaller grains. This race is in its critical period at the beginning of germination. After the germinant grains have once acquired a size much larger than the mean size of the smaller grains, the latter are absorbed readily at any temperature in the grain-growth region.

Another example of germinative temperature conditions is shown in Fig. 47, a micrograph of a molybdenum hook used to support tungsten filaments in electric incandescent lamps. This hook was in the fibrous condition when it was put in the lamp. It has been heated in the lamp from the heat of the tungsten filament which it supported and with which it was in contact at one point only, namely, at the bottom of the loop portion. It received heat by conduction and radiation from the coiled tungsten filament. There would be temperature gradients in two directions in this wire, one axially and one radially. The combination of these two temperature gradients has produced the structure seen in Fig. 47. It will be noted that at the point of contact between the tungsten wire and the molybdenum hook, the latter is fine grained. The temperature at this point was above the germinative temperature. At points more remote from the point of contact between the hook and the filament, there is every gradation in structure from fine grained throughout to coarse grained throughout. At the contact between the recrystallized and unrecrystallized portion (shown at the extreme right of Fig. 47) the molybdenum hook is very brittle. It is ductile in the fully recrystallized region and also in the unrecrystallized region.

#### EFFECT OF TIME AND TEMPERATURE ON GRAIN SIZE IN TUNGSTEN

A series of experiments was carried out on swaged tungsten rods 0.075 in. diameter made from tungsten metal of three different varieties. Two of the sets of samples contained 0.75 per cent. thoria and the third set contained no thoria. The results of these experiments are given in Table 3.

This table shows plainly that the increase in grain size with increase

in time is not regular. It is as regular, however, as might be expected with different samples. At the time these experiments were carried out, the writer was not aware of the extreme rapidity of the formation of germinant grains during heating. Some of the radical results indicated in Table 3 were found later to have been caused by the germinative temperature conditions during heating.

TABLE 3

Kind of Metal	Temperature °C.	Time of Exposure			Grains per Square Millimeter
		Hr.	Min.	Sec.	
(A) $\frac{3}{4}$ per cent. thoria....	2,500	0	0	30	3,400
$\frac{3}{4}$ per cent. thoria....	2,500	0	1	0	2,700
$\frac{3}{4}$ per cent. thoria....	2,500	0	5	0	2,700
$\frac{3}{4}$ per cent. thoria....	2,500	0	25	0	2,200
$\frac{3}{4}$ per cent. thoria....	2,500	2	0	0	3,000
$\frac{3}{4}$ per cent. thoria....	3,000	0	0	30	2,300
$\frac{3}{4}$ per cent. thoria....	3,000	0	1	0	2,200
$\frac{3}{4}$ per cent. thoria....	3,000	0	5	0	2,500
$\frac{3}{4}$ per cent. thoria....	3,000	0	25	0	1,800
$\frac{3}{4}$ per cent. thoria....	3,000	2	0	0	2,300
$\frac{3}{4}$ per cent. thoria....	3,000	9	45	0	2,200
(C) No thoria.....	2,500	0	0	30	1,700
No thoria.....	2,500	0	1	0	1,900
No thoria.....	2,500	0	5	0	2,800
No thoria.....	2,500	0	25	0	1,000
No thoria.....	2,500	2	0	0	1,600
No thoria.....	3,000	0	1	0	900
No thoria.....	3,000	0	5	0	1,500
No thoria.....	3,000	0	25	0	1,100
No thoria.....	3,000	2	0	0	1,900
(D) $\frac{3}{4}$ per cent. thoria....	2,500	0	0	30	2,000
$\frac{3}{4}$ per cent. thoria....	2,500	0	1	0	2,300
$\frac{3}{4}$ per cent. thoria....	2,500	0	5	0	1,700
$\frac{3}{4}$ per cent. thoria....	2,500	0	25	0	2,100
$\frac{3}{4}$ per cent. thoria....	2,500	2	0	0	1,800
$\frac{3}{4}$ per cent. thoria....	2,500	9	45	0	1,600
$\frac{3}{4}$ per cent. thoria....	3,000	0	0	30	1,900
$\frac{3}{4}$ per cent. thoria....	3,000	0	1	0	2,000
$\frac{3}{4}$ per cent. thoria....	3,000	0	5	0	1,900
$\frac{3}{4}$ per cent. thoria....	3,000	0	25	0	2,000
$\frac{3}{4}$ per cent. thoria....	3,000	2	0	0	1,700

The conclusions reached from the results given in Table 3 are as follows:

1. Recrystallization is complete in less than 30 sec. at temperatures of 2500° C. or above. Aside from the germinative temperature phenomena, grain growth has established an approximate equilibrium

grain size at the end of 30 sec. Further heating for  $9\frac{3}{4}$  hr. increases the grain size but slightly.

2. In the tungsten rods containing thoria, the difference in grain size between a temperature of  $2500^{\circ}\text{C.}$  and a temperature of  $3000^{\circ}\text{C.}$  with the time period constant is very slight.

3. With tungsten containing no thoria, the difference in grain size for a given time between  $2500^{\circ}\text{C.}$  and  $3000^{\circ}\text{C.}$  is more marked.

Tungsten ingots during sintering also attain an approximate equilibrium grain size for a given temperature in a few minutes. This is especially true at the higher temperatures of sintering, say  $3200^{\circ}\text{C.}$ , whereas if the sintering is done at a lower temperature, a longer time is necessary for an approximate equilibrium grain size to be produced.

#### STRUCTURE AND PROPERTIES OF TUNGSTEN FILAMENTS AFTER USE

Tungsten incandescent electric lamps are all so designed that when the lamps are used at rated voltage the temperature of the filaments will be above the recrystallization or equiaxing temperature of tungsten. This is especially necessary when the long time of heating of the filaments is considered. Since the operating temperatures of lamp filaments are in the grain-growth region of tungsten (above the equiaxing temperature and below the melting point) and since the time of heating in this grain-growth range is long, sometimes exceeding 1500 hr., the structure of the filament will change during the life of the lamp. After a short burning, tungsten wires containing thoria will possess structures similar to that shown in Fig. 48. It will be noted that the grains are longer in the direction of the working than in a perpendicular direction. During the life of the lamp, this elongation is even more marked. Fig. 49 is a micrograph of a 40-watt tungsten filament after burnout. This filament contained thoria. It will be noted that the grains have been greatly elongated during the life of the lamp. These grains are equilibrium grains so far as deformational strains are concerned. The growth in a longitudinal direction has been more rapid than in an axial direction. The underlying causes for this type of grain formation have been studied, and the following conclusions have been reached:

The spherical thoria globules existing in the tungsten ingot before working are elongated during the working operations. They do not, however, elongate as much as the metallic grains. The drawn tungsten wire will consist of metallic fibers of tungsten elongated in the direction of working and non-metallic fibers or miniature rods of thoria parallel to the metallic tungsten fibers. When this wire is heated above its equiaxing temperature, the metallic fibers will change into very small equiaxed grains, but the thoria rods, if they change at all, only break up into rows of spheres, each row occupying the same general position as the thoria rod from which it was formed. Even the breaking up of the

thoria into spheres takes considerable time. After the metallic grains of tungsten have broken up into very small equiaxed grains, these coalesce with one another, forming fewer and larger grains. Grain growth will take place in accordance with the time, temperature, and resistance to grain growth. The resistance to grain growth in an axial direction will be much greater than in a longitudinal direction. In an axial direction, the thoria rods, or rows of thoria spheres, will present much more surface to resist grain growth than in a longitudinal direction, because the sides of the thoria rods will offer resistance to radial grain growth. Only the ends of the thoria rods, however, will offer resistance to grain growth in a longitudinal direction. By calculation, it can be shown that the resistance to grain growth in an axial direction due to the presence of thoria may be more than 50 times that in a longitudinal direction.

It will be seen later that the forced formation of the elongated grains in a tungsten filament makes it very much more rugged than it would have been had the grains been exactly equiaxed. Fig. 50 is a micrograph of one of the old squirted or pressed tungsten filaments after life test. This micrograph was kindly furnished by Mr. C. D. Young, of the Pennsylvania Railroad Co. It will be noted that the grains in this wire are equiaxed. This is the structure which renders tungsten extremely brittle and fragile at ordinary temperatures, whereas the structure shown in Fig. 49 renders tungsten rugged at room temperature. The reasons for this will be discussed later.

A great many different types of structure are encountered in tungsten filaments after they have been used for various lengths of time. Fig. 49 is a typical example.

The difference in resistance to grain growth between tungsten containing thoria and tungsten containing no thoria can be seen by referring to Figs. 51 and 52. Fig. 51 contains 0.75 per cent. thoria and Fig. 52 no thoria. Both wires have been heated for 10 min. to a temperature near 2850° C. These micrographs are of the natural surfaces of the wires. The boundary material of the grains volatilizes more rapidly than the crystalline material, so that the grain boundaries can be seen without etching.

If tungsten develops very coarse grains when used as a filament, it has the property of being much stiffer at high temperatures than when composed of fine grains. The wires will not sag due to their own weight even when heated to a temperature of about 2800° C. If the filament, however, is fine grained, it may sag badly due to its own weight. There is danger in producing coarse-grained structures, because if a boundary line between two grains cuts sharply across a section of the filament on a plane approximately perpendicular to its axis, one section of the filament may be displaced in a direction perpendicular to its axis, thus causing the phenomenon known as "offsetting." We now know that at

the boundaries between two tungsten grains a film of amorphous tungsten exists (this assumes the validity of the amorphous theory) and that at the temperatures at which the lamp filaments are used, this amorphous film is more or less fluid and weak mechanically. This is why the fine-grained tungsten wires are weaker at high temperatures than the coarse-grained tungsten. This is also the reason that offsetting takes place at the grain boundaries traversing the whole cross-section of the wire.

It is possible to form tungsten wires which are both coarse grained and substantially non-offsetting. Fig. 53 is a micrograph of a tungsten filament that is both coarse grained and non-offsetting. It can be noted that these grains are very long in a direction parallel to the axis of the wire, and that at no place does a grain boundary extend across the diameter of the wire in such a way that offsetting can take place.

#### WELDABILITY OF TUNGSTEN

Many attempts have been made to weld the particles of tungsten together by working at a very high temperature. No headway has been made in this direction. To be workable, a piece of tungsten must be substantially non-porous—that is, the individual particles must have been previously welded together at a temperature near the melting point of tungsten in an atmosphere of hydrogen or other gas which is either helpful or at least not harmful to the tungsten.

Sometimes the swaged tungsten rods split during the swaging operation. Attempts have been made to weld these split portions together by working at a high temperature. The highest temperature available was 1700–1800° C. It was not found possible to weld the tungsten in this manner. Tungsten can be welded electrically at temperatures near fusion.

#### NOTES ON POLISHING, MOUNTING AND ETCHING TUNGSTEN

Tungsten is not an easy metal to polish. It is so resistant to the action of abrasion of the polishing powders that levigated alumina can be substituted for tripoli to advantage just preceding the rouge.

White cast iron has been used to advantage as a mounting material for small pieces of tungsten which cannot conveniently be handled without some sort of a mounting. The piece of tungsten to be mounted is put in a mold and the molten cast iron poured around it. The white cast iron and tungsten are so nearly the same hardness that flat surfaces can be produced on the tungsten during polishing. It is sometimes very difficult to mount and polish the smallest tungsten wires. This has been accomplished in a successful manner, however, on wires less than 0.001 in. in diameter. One satisfactory method is as follows: An ordinary malleable iron  $\frac{3}{8}$ -in. (9.5-mm.) pipe cap is planed on the closed end outside. It is then drilled on the inside to a plane parallel to the outside plane. A round cover glass is put on the inside of the pipe cap, on which are placed

several pieces of the small tungsten wire to be polished. Another cover glass is placed on top of these wires, after which the opening of the pipe cap is filled with powdered glass. It is then put in a furnace, being maintained in an upright position, and heated for about 5 min. to a temperature of 800° or 900° C. It has been found in many experiments that this temperature does not affect the structure of tungsten wire. The pipe cap with contents is then removed from the furnace and the glass which has congealed is pressed tightly into the pipe cap and allowed to cool slowly. The metal portion on the end of the pipe cap is then turned off in a lathe and the glass is exposed. This glass containing the samples of tungsten is polished in the ordinary manner until the tungsten wires are exposed, and the polishing is completed in the ordinary manner.

Boiling hydrogen peroxide is used for etching most of the tungsten products. Tungsten may also be etched electrolytically with good results, using a solution of sodium hydrate for electrolyte.

#### WHY IS FIBROUS TUNGSTEN DUCTILE?

This question has been the subject of much work and much thought. The fact that fibrous tungsten is ductile has been known for 10 years, but no explanation has been forthcoming. The result could not have been predicted from metallurgical knowledge. The explanation has been worked out step by step. The researches which have finally resulted in an explanation also permit the postulation of some new metallographic laws relating to all metals. In making the explanation, the amorphous theory is considered valid. The explanation, however, does not depend upon the validity of the amorphous theory or any other theory; it depends on facts which have been experimentally ascertained. These researches may, in fact, be used to strengthen the amorphous theory.

Crystalline tungsten is somewhat malleable and ductile at room temperature—that is, if a single grain of tungsten can be isolated, it can be slightly deformed cold before rupturing. The cold deformation strain hardens the tungsten grain and makes it brittle, similar to this action in common ductile metals. Crystalline tungsten can also be deformed above room temperature; it is capable of more permanent deformation without becoming brittle, the higher the temperature at which the deformation is produced.

Amorphous tungsten is very brittle at room temperature. It is capable of being deformed without rupture at high temperature. Tungsten composed of small equiaxed grains is not ductile or malleable at room temperature. It may be both malleable and ductile at elevated temperatures. A fracture at room temperature shows that the break has taken place largely at the grain boundaries. Occasionally, along the fracture line, a grain is encountered which has been broken in two, but this will be



caused by a local high resistance to rupture along the grain boundaries. Fine-grained tungsten can be deformed under suitable conditions (such as are outlined previously) at high temperatures.

The reasons why fibrous tungsten is ductile when cold can be stated briefly as follows: Tungsten composed of equiaxed grains is brittle at room temperature because the brittle amorphous phase at the grain boundaries permits rupture before a load sufficient to deform the malleable crystalline phase can be applied. Tungsten possessing this structure is brittle even though the crystalline material present is more capable of permanent deformation at room temperature than the crystalline portions of fibrous tungsten. Fibrous tungsten is ductile at room temperature (assuming that the fiber has been produced under proper conditions) in spite of the fact that it contains more of the amorphous phase than equiaxed tungsten. It is ductile because the grain distortion by working arranges the grain boundaries in such a manner that the resistance to rupture along them is so great that rupture is forced to take place through the deformed grains themselves. These grains, which will usually have been deformed above room temperature, will possess the ability to be further deformed at room temperature; rupture through them cannot take place without further deforming them, so the metal assumes ductility. The subject of deformation of metallic grains at a certain temperature and the increase of ductility by lowering the temperature will be treated later.

We must accept as a fact, which I have observed many times, that rupture along the amorphous planes in tungsten is much more pronounced along grain boundaries than along the amorphous slip planes in deformed tungsten grains. It is probable that the path of rupture along the amorphous slip planes in a deformed grain would be many times more intricate, involving intermeshings of submicroscopic size, than the path of rupture along the grain boundaries themselves. Whatever may be the explanation for this, we must accept it as fact. It should be kept in mind that only when the amorphous phase of the metal becomes very brittle does the fracture seek the grain boundaries. This condition obtains in tungsten at room temperature but not in the ordinary ductile metals.

Even a fibrous tungsten wire which is ductile is only malleable when the deforming pressure is applied at all points of its circumference at the same time, like the swaging die action. When one of these wires is placed on an anvil and struck with a hammer, it splits into many threads—that is, the deformed grains separate from each other.

Probably one of the major causes for rupture along the amorphous grain boundaries in tungsten, in preference to through the grains themselves, is the difference in coefficient of expansion of the amorphous and crystalline phases. Glass, a completely amorphous substance, can be

cracked by unequal heating or cooling, the cracks resulting from the different degrees of expansion or contraction of different parts of the piece. If we consider that fine-grained tungsten is made up of crystalline grains surrounded by films of amorphous tungsten, then these two phases will have different coefficients of thermal expansion. At the higher temperatures, these differences are easily adjusted because of the plasticity of the amorphous phase. At lower temperatures, however, both the amorphous and crystalline phases will be very rigid. This will force the strains of unequal expansion during heating or unequal contraction during cooling, into either the crystalline or amorphous material, or both. There are several reasons why these strains at low temperatures should be taken up largely by the amorphous phase. The amorphous phase is the only one possessing continuity. The crystalline phase consists of grains none of which actually touch each other. The continuity of the system depends on the amorphous phase. Any difference in coefficient of expansion between the amorphous and crystalline phases, no matter in what direction the difference may be, must affect therefore the amorphous phase. If internal strains are set up in this manner at the grain boundaries, smaller external loads will be required to cause rupture than would be indicated by the actual measure of cohesion of the isolated amorphous phase.

To obtain a somewhat better idea of these phenomena, let us consider the properties of tungsten wires about 0.007 in. (0.18 mm.) diameter with four types of structure.

1. *The Whole of the Wire Is Composed of a Single Grain.*—Since crystalline tungsten is somewhat malleable and ductile at room temperature, such a wire could be deformed cold. Cold deformation would strain-harden the tungsten and make it more brittle, and when continued far enough would break it because of the brittleness. The permanent deformation of the crystalline tungsten would generate amorphous tungsten at the planes of slip. The hardening and embrittling would be caused by the amorphous metal.

2. *The Tungsten Wire is Composed of Small Equiaxed Grains.*—Such a tungsten wire is brittle and fragile at room temperature. It cannot be appreciably bent (except the bending which takes place within its elastic limit) without breaking. The break will take place largely along the grain boundaries. These grain boundaries consist of thin films of tungsten in the brittle amorphous condition. The amorphous phase has in reality greater cohesion than the crystalline phase at room temperature, but it is under great internal stress due to the difference in coefficient of expansion between it and the crystalline phase.

3. *The Tungsten Wire Has a Fibrous Structure.*—To obtain a mental picture of this particular structure, suppose the ingot from which the wire was made was  $\frac{1}{4}$  in. (6.35 mm.) square and contained 3800 grains per

square millimeter. It is worked at temperatures below that of equiaxing so that the grains are progressively elongated from the beginning of working. When the wire is 0.007 in. diameter, the grains will have been changed into fibers the average length of which will be about 1 in. and the average diameter about 0.00002 in. The end of a given metallic fiber will usually not be contiguous to the ends of other fibers with which it is in contact. A tungsten wire with such a structure is ductile cold. It can be drawn cold, bent cold, coiled, etc. It will contain more amorphous tungsten than the fine-grained sample which was brittle. It will be ductile cold because the metal has a tendency to break along the amorphous planes at the grain boundaries and the path of rupture along these will be so great that the break is forced to take place through the crystalline material which, though not as malleable and ductile as the crystalline material before strain hardening, will still possess the properties of malleability and ductility to a certain extent at room temperature. A break through it, therefore, must cause a certain amount of deformation before rupture can take place, thus giving rise to the property of ductility.

4. *The Wire Consists of Elongated Grains Which Have Not Been Strain-hardened.*—Such a structure results from long heating of tungsten containing thoria or other non-metallic substances. The arrangement of the grains makes a long path for rupture along the grain boundaries, so a much greater load can be applied before rupture than when the metal is composed of equiaxed grains. This makes tungsten possessing an elongated grain structure stronger or more rugged than the equiaxed structure. At times, such wires even possess slight ductility at room temperature. Electric incandescent lamp filaments frequently possess these elongated grain structures and are as a consequence very rugged.

It is thus seen that the ductility of tungsten at room temperature does not depend on the quantity of amorphous tungsten, but on its arrangement. It has been stated before that the tensile strength of a tungsten ingot is about 18,000 lb. per square inch. Tungsten wire possessing a fibrous structure at 0.007 in. diameter will have a tensile strength of about 340,000 lb. per square inch. The actual cohesion is greater in the drawn tungsten wire than in the equiaxed ingot. The cohesion measured in each case is that of a system made up of what may be considered physically as two separate materials with different properties, but in the case of the equiaxed ingot the internal weakening strains predominate and in the case of the fibrous wire they are eliminated.

As an example of the action of the crystalline and amorphous phases in equiaxed and fibrous tungsten, let us suppose that a structure is made up of grains of iron which we will consider as analogous to the crystalline tungsten, the grains being bound together with very thin films of glass which we will consider analogous to the amorphous tungsten cement surrounding the grains. In the first example, let us suppose that the

grains of iron are equiaxed and unstrained—that is, similar to equiaxed tungsten. This structure will assume largely the properties of the brittle glass cement. If a grain of iron could be isolated, it would be malleable, but if the structure taken as a whole is hammered, it will fly to pieces, and if broken in tension, the breaking load will be too small to force any marked permanent deformation on the iron grains. This structure will be brittle. If, however, the structure were heated to a red heat, at which temperature both iron and glass are known to be workable, then considerable deformation could be effected by hammering or rolling. Let us suppose that such a structure could be rolled or drawn while hot until the dimensions of the deformed iron grains would be comparable to the dimensions of tungsten fiber. The glass film surrounding the grains of iron will remain intact. (In the actual working of the metal, it is very probable that the amorphous films surrounding the grains are made thicker by mechanical working.) Let us further suppose that in this amount of deformation the iron grains still possess some ductility in the cold. When cold ductility tests are made, it will be found that the glass films are no longer in a commanding position and the path of rupture will be forced largely through the deformed iron grains themselves. Such a structure with the same quantity of glass present will be ductile at room temperature. It has not been attempted to make the analogy perfect, because glass does not possess the same properties as amorphous tungsten, nor does iron possess the same properties as crystalline tungsten, and glass could not be generated within an iron grain during deformation.

That the fracture in tungsten tends to take place along the grain boundaries is shown conclusively in Fig. 55. This fracture was in a coarse-grained sample. One of the cracked boundary lines in Fig. 55 extends to the surface of the tungsten ingot, and the crack does also. In one place where the grain boundary line was rather jagged, the fracture took place through a portion of the crystalline material rather than at the grain boundary. The other cracked grain boundary abuts a fine-grained portion of the tungsten ingot at which the crack stops abruptly. This shows that resistance to rupture is less along a rather straight grain boundary than around the grain boundaries of fine-grained metal. Fig. 56 shows how the crack has jumped across the crystalline portion in preference to following the jagged grain boundary line. Fig. 57 shows the abrupt ending of a crack where the grain boundary line between two large grains intersects a fine-grained area, and Fig. 58 shows that the fracture of a fine-grained area follows in general the grain boundary line. There are two or three cracks in Fig. 42, and it can be plainly seen that these cracks follow the grain boundaries.

That rupture tends to take place along the boundaries of deformed grains in fibrous tungsten can be seen from Fig. 54. That the fracture

does not take place entirely along these grain boundaries is due to the fact that the resistance to rupture through the deformed grains or fibers is less in fibrous tungsten than along the deformed grain boundaries. If the deforming load is applied to fibrous tungsten longitudinally, the wire is ductile, but if applied transversely, it is brittle.

#### SOME GENERAL METALLOGRAPHIC PROPOSITIONS

The discussion in the preceding caption shows why fibrous tungsten is more ductile at room temperature than equiaxed tungsten. Another significant fact has been observed with tungsten, namely, that after the limit of ductility has been reached by working at, say, a red heat, the tungsten after cooling to room temperature or other lower temperatures becomes ductile at these temperatures. Experiments have been made which show that this phenomenon is common to all ductile metals. The reasons have been ascertained. The underlying reason for the loss of ductility by working a metal at a certain temperature below its annealing temperature and the regaining of ductility by cooling to some lower temperature, is that the amorphous phase of any metal will increase in cohesion on cooling, at a faster rate than the crystalline phase. Let us refer to Plate 3. This shows the general cohesion-temperature curves of the amorphous and crystalline phases of metals. The direction of the curves will have to be determined for any given metal, but the general relationship will be found similar to those given in Plate 3. The curve traced in by a continuous line represents the change in cohesion of the amorphous phase with change in temperature. The cohesion is substantially zero at the melting point of the metal and increases as the temperature decreases, reaching a maximum at absolute zero. The dotted curve represents the change in cohesion of the crystalline phase with change in temperature. The crystalline phase disappears and changes into the amorphous phase when the metal is melted. On cooling from above the melting point, the crystalline phase forms during solidification and immediately at the melting temperature its cohesion assumes a finite value many times greater than that of the amorphous phase at the same temperature. On cooling below the melting point, however, the crystalline phase increases in cohesion at a very much slower rate than the amorphous phase. At some temperature between the melting point and absolute zero (in most metals not far from 0.35 to 0.45 of the absolute melting point) the cohesion of the crystalline phase will be the same as that of the amorphous phase. This temperature I have called the "equi-cohesive temperature."<sup>34</sup> It corresponds in most, if not all, metals, to the lowest equiaxing temperature of the severely cold-worked metal. Just as the equiaxing temperature of the metal is increased with decrease in the time of heating, the apparent equi-cohesive temperature increases as the

time of applying the load by which the cohesion is measured is decreased. If a metal is deformed above the equi-cohesive temperature and kept at that temperature, the grains will not remain permanently deformed, but will equiaxe; the properties of the metal will be different after equiaxing. If, however, the grains are deformed below the equi-cohesive temperature, they will remain permanently distorted. Below the equi-cohesive

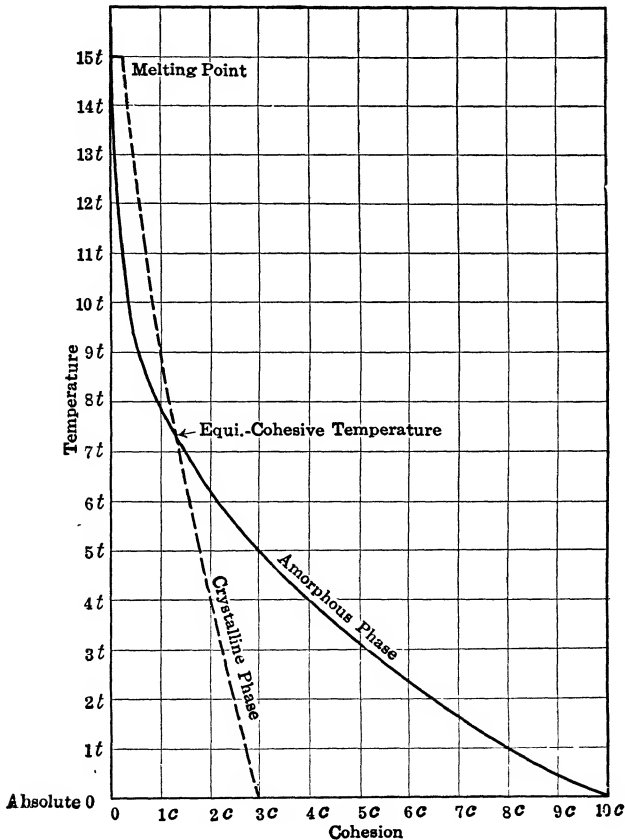


PLATE 3.—COHESION-TEMPERATURE CURVES OF AMORPHOUS AND CRYSTALLINE PHASES OF ANY METAL.

temperature, the amorphous phase has not only greater cohesion than the crystalline phase but its cohesion increases much faster with decrease in temperature.

Let us suppose that Plate 3 represents the cohesion-temperature curves of tungsten and that the temperature of wire drawing is  $4t^{\circ}$ . The crystalline phase at this temperature has a cohesion of  $2c$  and the amorphous phase  $4c$ . The wire drawing can be continued until the load necessary to break the wire at a temperature of  $4t^{\circ}$  is not sufficient to

deform the crystalline phase further. Before any deformation has taken place at  $4t^\circ$  the crystalline phase will have a cohesion of  $2c$ , but as the deformation continues amorphous metal will be generated at the planes of slip, and these being in a commanding position will increase the apparent cohesion of the deformed grain. Now let us suppose that room temperature corresponds to  $1t^\circ$ . The cohesion of the crystalline phase at this temperature is  $2.7c$  and that of the amorphous phase is  $8.1c$ . It will be noted that the amorphous phase at room temperature is  $5.2c$  more cohesive than the crystalline phase, whereas at  $4t^\circ$  it is only  $2c$  more cohesive. The limit of ductility at  $4t^\circ$  was governed to a certain extent by the difference in cohesion between the amorphous and crystalline phases. By cooling to room temperature this difference has increased  $3.2c$ . If now the metal is subjected to a ductility test at room temperature, a load sufficient to deform the crystalline phase further can be applied before rupture takes place; in other words, the metal is ductile and it has gained ductility because the amorphous phase increases in cohesion with decrease in temperature at a faster rate than the crystalline phase.

One point must not be confused. The limit of ductility of a metal at a certain temperature does not mean that the crystalline phase has been deformed to the greatest possible extent at that temperature. It simply indicates that the load necessary to deform the crystalline phase further cannot be applied to the metal by tension without breaking it. Even though it takes a greater load to deform the crystalline phase at a lower temperature, the increased load which can be applied by tension before rupture takes place (depending largely upon the cohesion of the amorphous phase) will be much more than the increase in the cohesion of the crystalline phase. Much more deformation can be forced on a given metal at a temperature below that of equiaxing by application of pressure than by tension. Rupturing of metals by pressure, however, must be considered as a natural modification of rupture by tension. A metal, for example, can be subjected to the highest hydrostatic pressures now available without permanently deforming it in the least. Pressure alone, therefore, will not change the external shape of a metal—that is, it will not permanently deform it. Deformation by pressure, therefore, implies a difference in pressure between two parts greater in magnitude than the cohesion of the metal to be deformed. In some cases, during the application of pressure to a metal, certain parts are actually in a state of tension and may break because of it. In other cases all parts of the metal may be subjected to pressure but certain parts will receive enough more pressure than other parts to cause permanent deformation. By utilization of the property of malleability, a metal may be deformed to a greater extent than by the utilization of ductility.

It is doubtful whether the actual limit of deformation of any crystalline metal has ever been reached experimentally. The temporary limit of deformation is always reached when the load necessary to cause it also causes the metal to rupture.

Beilby's observations on copper, gold, and silver fit in with these ideas very nicely. A description of his experiments are here given in his own words:\* "Gold, silver and copper of the highest purity were the metals used. The diameters of the wires at all stages were carefully measured by a micrometer screw gage. The increases of length by wire drawing were also measured. After a final annealing, wires were drawn to as much as fourteen times their original length. Their tenacity was determined by applying a water load to the vertically hung wire. As stated above, the maximum tenacity was reached in wires drawn from three to five times their original length. A hard-drawn wire shows no general extension even under the breaking load. The broken pieces when brought together show that the small local extension which occurs at the point of rupture may amount to 1 per cent., or less, but it is purely local as proved not only by the absence of any general stretching, but also by a full series of measurements of the diameter at many points along the length. The tenacity was always calculated on the average cross-sections worked out from these measurements. After a large number of experiments had been made at the ordinary temperature, it was decided to repeat these at the temperature of liquid air or  $-182^{\circ}$ . The results showed that at this low temperature the tenacity of these metals is very much increased, gold rising from 15.6 tons per square inch at  $15^{\circ}$  to 22.4 tons at  $-182^{\circ}$ , silver from 25.7 tons at  $15^{\circ}$  to 34.4 tons at  $-182^{\circ}$ , and copper from 28.4 tons at  $15^{\circ}$  to 36 tons at  $-182^{\circ}$ . The most unexpected result was that all the hard-drawn wires stretched 11 to 12 per cent. at the lower temperature before breaking. The plasticity which had disappeared at  $15^{\circ}$  reappeared at the lower temperature. Our conclusion at the time was that the restarting of plasticity at the low temperature was due to the general increase of tenacity which enabled the wire better to resist the disruptive strains in the die."

Our discussions show that the mere increase in tenacity cannot be responsible for the ductility which Beilby observed. For example, Hadfield<sup>38</sup> found that the ductility of iron decreased with increased tenacity as the temperature was lowered to that of liquid air. What happened in Beilby's experiments was that the tenacity increased faster with decrease in temperature than the cohesion of the crystalline phase due to the differential cohesion between it and the amorphous phase. In Plate 3, let us suppose that room temperature corresponds to  $3t^{\circ}$  and liquid air temperature to  $1t^{\circ}$  and the curves as given represent in general the prop-

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\* *Journal, Institute of Metals* (No. 2, 1911) 6, 19.



erties of the crystalline and amorphous phases of the metals with which Beilby experimented. At room temperature the cohesion of the crystalline phase is  $2.2c$ , and of the amorphous phase  $5.1c$ , while at  $1t^\circ$  the cohesions are  $2.7c$  and  $8.1c$  respectively. Beilby had cold-drawn the wires at room temperature till they were brittle. On cooling to  $-182^\circ$  ( $1t^\circ$ ), the ratio between the cohesion of the amorphous and crystalline phases had greatly increased so that at  $1t^\circ$  tensile load could be applied before the metals ruptured sufficiently to deform the crystalline phase further.

It will be evident, from what has been said, that the metals might be wire drawn at  $1t^\circ$  till they became brittle at that temperature and they would then become ductile if cooled to a temperature of, say,  $\frac{1}{2}t^\circ$ .

A further experiment with a wire of aluminum copper alloy also fits in nicely with these fundamental ideas. The wire was worked cold at room temperature until it would break when bent through an angle of about  $60^\circ$ . A piece of wire was then immersed in liquid air and was bent double and straightened again without cracking.

Further confirming evidence is at hand and a rather complete set of experiments dealing with these general problems has now been completed and will be reported in a separate paper. The hypothesis has been sufficiently verified so that results can be predicted from it.

I have considered the application of this hypothesis to substantially pure metals and solid solutions. The application to other classes will be more complicated. Let us take steel as an example. The cohesion-temperature curves of pure iron have jogs in the crystalline curve corresponding to the allotropic points. The approximate curves for iron as well as for cementite would have to be ascertained. In annealed steel, at least three physically different substances will be present; namely, crystalline ferrite, crystalline cementite and the amorphous solution of iron and iron carbide. After cold deformation there may be five physically different constituents present; namely, crystalline ferrite, amorphous iron, crystalline cementite, amorphous cementite and the amorphous solution of iron and cementite. By knowledge of the properties of each of these constituents and their structural positions in the steel, predictions may be made regarding the physical properties of the piece of steel taken as a whole.

It should be pointed out that the metals which have the greatest possibilities for improvement by mechanical working, or those metals which should ultimately have high tensile strength whether coupled with ductility or not, should (a) possess high unit cohesion in both the crystalline and amorphous phases, and (b) the cohesion of the latter should be much greater than that of the former at room temperature. Such metals will have relatively high equi-cohesive temperatures.

In fibrous tungsten, a condition is obtained which makes the ductility

greater at room temperature than that of the equiaxed tungsten. The general hypothesis of increase in ductility in metals already possessing fibrous structures by decreasing the temperatures is entirely independent of the relative ductility of the fibrous and equiaxed structures. In Beilby's experiments quoted above, for example, the ductility at  $-182^{\circ}$  of the metals with fibrous structures may not have been as great as that of the equiaxed metals at the same temperature. With most, if not all, metals, however, some low temperature will be reached at which the metals in the equiaxed condition will be brittle and the same metals possessing fibrous structures which will have been produced at some higher temperature will be ductile. Iron, for example, possesses these properties at the temperature of liquid air, and tungsten and molybdenum, at ordinary or room temperature.

The amorphous phase of iron is supposed to be brittle at room temperature. It is not sufficiently brittle to cause rupture along the grain boundaries at room temperature. Liquid air temperature, however, will cause coarse-grained iron to fracture along the grain boundaries. A metal with an equiaxed grain structure cannot be very ductile at high temperature because the amorphous phase is weaker than the crystalline. Its ductility (assuming one of the ductile metals) increases on cooling to a certain point, below which it begins to become less ductile. The temperature may be lowered sufficiently to make it brittle.

Experimental proof has also been obtained showing that if a metal is worked at a certain temperature below its annealing temperature, till it becomes brittle at that temperature, it is more brittle at higher temperatures below that of annealing.

#### ACKNOWLEDGMENTS

I wish to express my indebtedness to B. L. Benbow, Manager of the Cleveland Wire Division of the General Electric Co., who instigated the work on the metallography of tungsten and under whose direction the work was carried out. I wish also to thank Messrs. L. Cover, L. S. Twomey, E. J. Hull, W. T. Burgoon, and W. P. Sykes, for valuable aid in carrying out the experiments.

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FIG. 1.—TUNGSTEN INGOT  $\times 152$ .



FIG. 2.—SWAGED TUNGSTEN ROD,  
0.214 IN. DIAM.  $\times 152$ .



FIG. 3.—SWAGED TUNGSTEN ROD, 0.125 IN. DIAM.  $\times 152$ .



FIG. 4.—SWAGED TUNGSTEN ROD,  
0.082 IN. DIAM.  $\times 152$ .

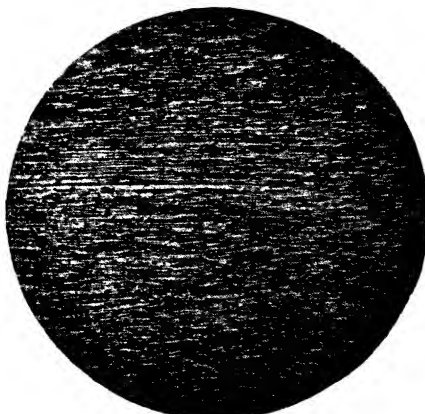


FIG. 5.—SWAGED TUNGSTEN ROD,  
0.030 IN. DIAM.  $\times 152$ .

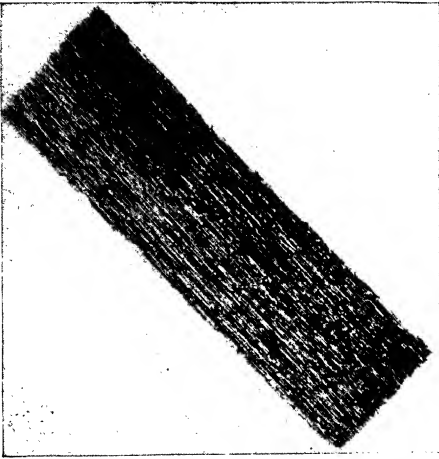


FIG. 6.—DRAWN TUNGSTEN WIRE, 0.010 IN. DIAM.  $\times 169$ .

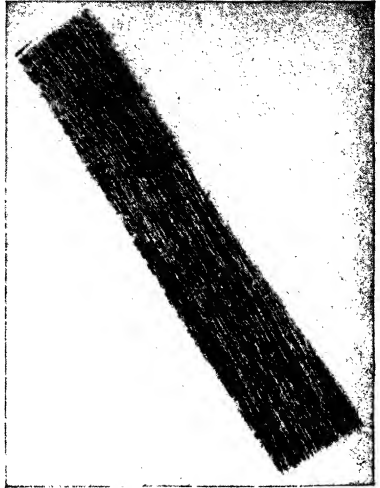


FIG. 7.—DRAWN TANTALUM WIRE, 0.007 IN. DIAM.  $\times 125$ .



FIG. 8.—TANTALUM WIRE ANNEALED 5 MIN. AT 1600° C. IN VACUUM.  $\times 125$ .



FIG. 9.—TANTALUM WIRE ANNEALED 1 MIN. IN HYDROGEN ATMOSPHERE.  $\times 125$ .

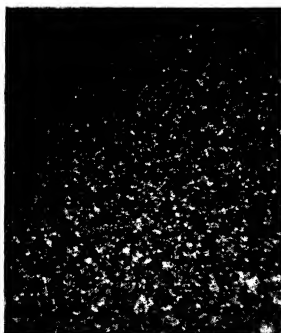


FIG. 10.—CONTAINING  
1 PER CENT.  $\text{ThO}_2$ .

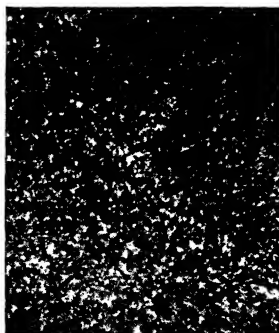


FIG. 11.—CONTAINING  
2 PER CENT.  $\text{ThO}_2$ .



FIG. 12.—CONTAINING  
3 PER CENT.  $\text{ThO}_2$ .

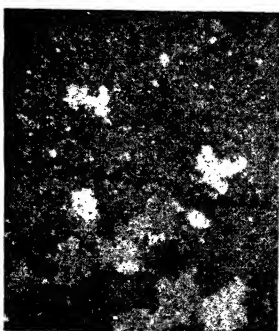


FIG. 13.—CONTAINING  
4 PER CENT.  $\text{ThO}_2$ .

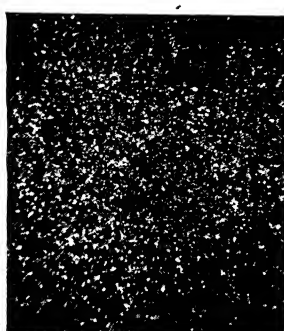


FIG. 14.—CONTAINING  
5 PER CENT.  $\text{ThO}_2$ .

FIG. 10 TO 14.—TUNGSTEN INGOTS HEATED 12 MIN. AT  $3200^\circ\text{C}$ .  $\times 16$ .

FIG. 15.—CONTAINING 1 PER CENT.  $\text{ThO}_2$ .FIG. 16.—CONTAINING 2 PER CENT.  $\text{ThO}_2$ .FIG. 17.—CONTAINING 3 PER CENT.  $\text{ThO}_2$ .FIG. 18.—CONTAINING 4 PER CENT.  $\text{ThO}_2$ .FIG. 19.—CONTAINING 5 PER CENT.  $\text{ThO}_2$ .

FIG. 15 TO 19.—TUNGSTEN INGOTS HEATED 12 MIN. AT  $3200^\circ\text{C}$ .  $\times 389$ .





FIG. 20.—TUNGSTEN INGOT SHOWING TWO LARGE GRAINS IN POSITION TO ABSORB SMALL GRAINS.  $\times 117$ .

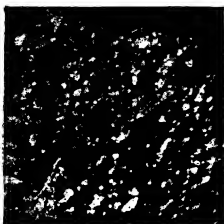


FIG. 21.

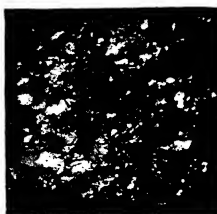


FIG. 22.

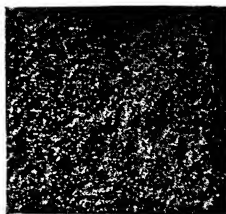


FIG. 23.

FIG. 21.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ \text{C}$ .  $\times 5$ .

FIG. 22.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 30 MIN. AT  $2600^\circ \text{C}$ .  $\times 5$ .

FIG. 23.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ \text{C}$ . AND HELD FOR 30 MIN.  $\times 5$ .

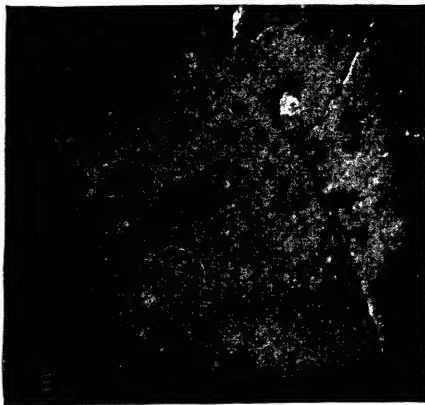


FIG. 24.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN.



FIG. 25.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 30 MIN.



FIG. 26.—LONGITUDINAL SECTION AT SECTION A-A OF FIG. 25.  $\times 9$ .

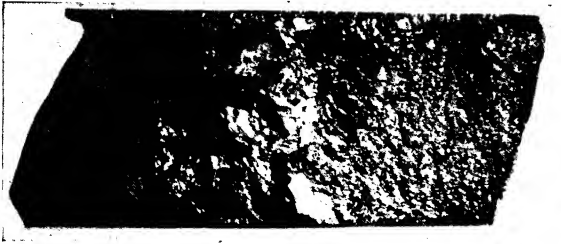


FIG. 27.

FIG. 27.—LONGITUDINAL FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ . COLD END IN ELECTRODE CLAMP.  $\times 5$ .

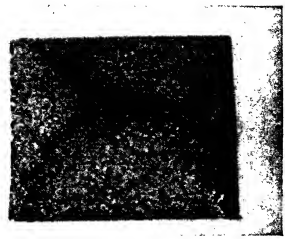


FIG. 28.

FIG. 28.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ\text{C}$ . AND HELD 30 MIN.  $\times 5$ .

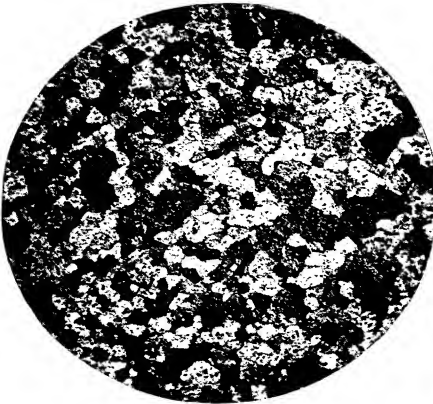


FIG. 28a.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ\text{C}$ . AND HELD 30 MIN.  $\times 144$ .

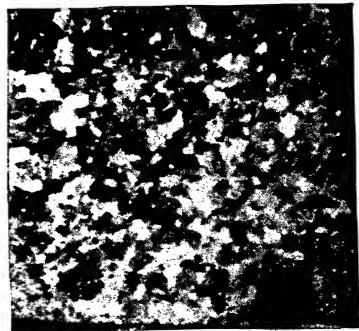


FIG. 29.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ\text{C}$ . AND THEN HEATED 10 MIN. AT  $3200^\circ\text{C}$ .  $\times 7$ .

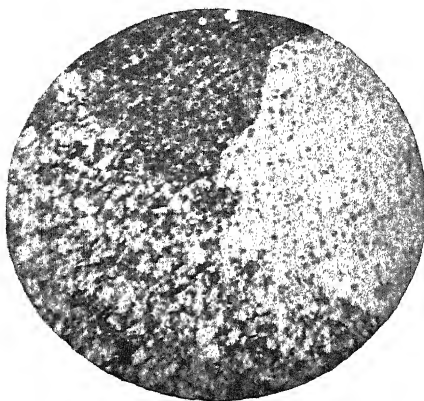


FIG. 20.—TUNGSTEN INGOT SHOWING TWO LARGE GRAINS IN POSITION TO ABSORB SMALL GRAINS.  $\times 117$ .



FIG. 21.

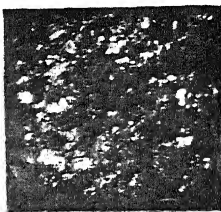


FIG. 22.

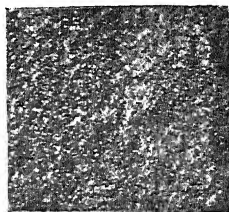


FIG. 23.

FIG. 21.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ\text{C}$ .  $\times 5$ .

FIG. 22.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 30 MIN. AT  $2600^\circ\text{C}$ .  $\times 5$ .

FIG. 23.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ\text{C}$ . AND HELD FOR 30 MIN.  $\times 5$ .

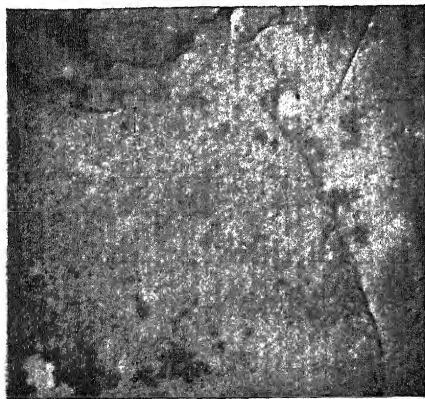


FIG. 24.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ\text{C}$ . TRANSVERSE SECTION.  $\times 9$ .



FIG. 25.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 30 MIN. AT  $2600^\circ\text{C}$ . TRANSVERSE SECTION.  $\times 9$ .



FIG. 26.—LONGITUDINAL SECTION AT SECTION A-A OF FIG. 25.  $\times 9$ .

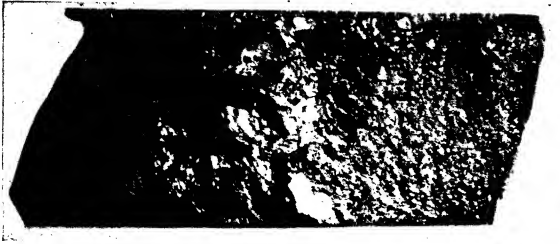


FIG. 27.

FIG. 27.—LONGITUDINAL FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ . COLD END IN ELECTRODE CLAMP.  $\times 5$ .



FIG. 28.

FIG. 28.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ \text{C}$ . AND HELD 30 MIN.  $\times 5$ .

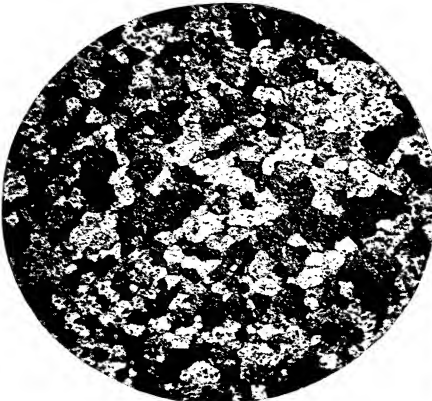


FIG. 28a.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ \text{C}$ . AND HELD 30 MIN.  $\times 144$ .

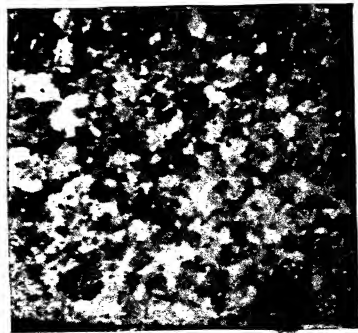


FIG. 29.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ \text{C}$ . AND THEN HEATED 10 MIN. AT  $3200^\circ \text{C}$ .  $\times 7$ .



FIG. 20.—TUNGSTEN INGOT SHOWING TWO LARGE GRAINS IN POSITION TO ABSORB SMALL GRAINS.  $\times 117$ .

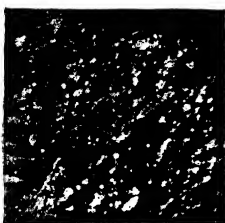


FIG. 21.

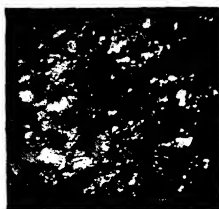


FIG. 22.

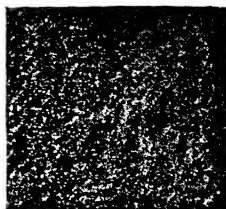


FIG. 23.

FIG. 21.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ \text{C}$ .  $\times 5$ .

FIG. 22.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 30 MIN. AT  $2600^\circ \text{C}$ .  $\times 5$ .

FIG. 23.—TRANSVERSE FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ \text{C}$ . AND HELD FOR 30 MIN.  $\times 5$ .

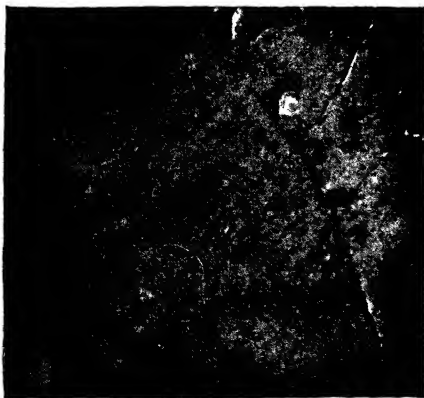


FIG. 24.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ \text{C}$ . TRANSVERSE SECTION.  $\times 9$ .

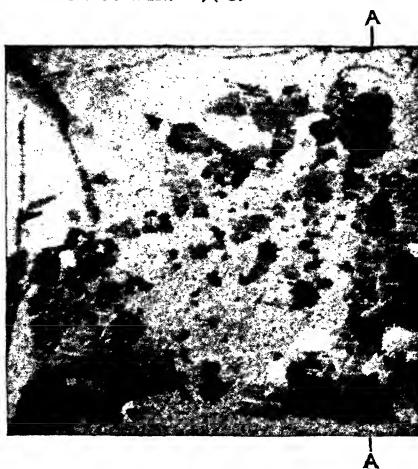


FIG. 25.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 30 MIN. AT  $2600^\circ \text{C}$ . TRANSVERSE SECTION.  $\times 9$ .



FIG. 26.—LONGITUDINAL SECTION AT SECTION A-A OF FIG. 25.  $\times 9$ .

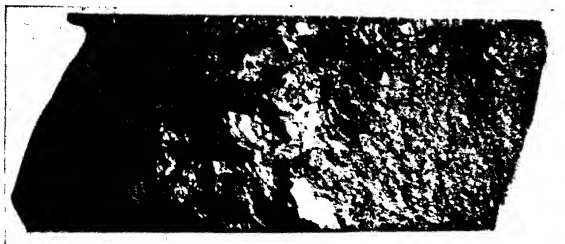


FIG. 27.

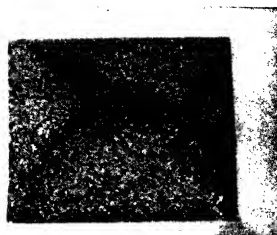


FIG. 28.

FIG. 27.—LONGITUDINAL FRACTURE OF TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ . COLD END IN ELECTRODE CLAMP.  $\times 5$ .

FIG. 28.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ \text{C}$ . AND HELD 30 MIN.  $\times 5$ .

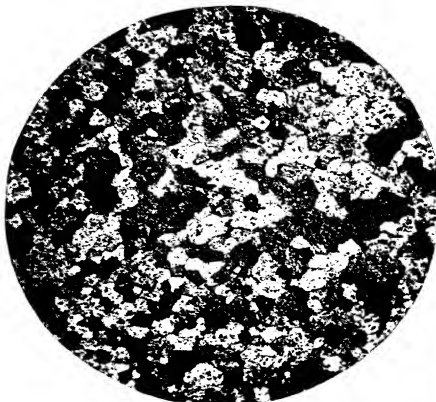


FIG. 28a.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED QUICKLY TO  $3200^\circ \text{C}$ . AND HELD 30 MIN.  $\times 144$ .

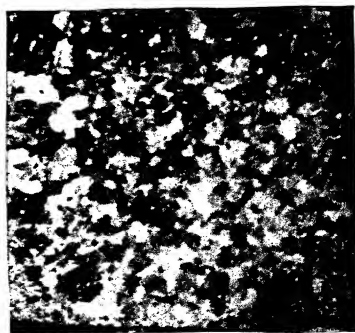


FIG. 29.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ \text{C}$ . AND THEN HEATED 10 MIN. AT  $3200^\circ \text{C}$ .  $\times 7$ .



FIG. 29a.—TUNGSTEN INGOT CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , HEATED 20 MIN. AT  $2600^\circ\text{C}$ . AND THEN HEATED 10 MIN. AT  $3200^\circ\text{C}$ .  $\times 74$ .



FIG. 30.—FINE-GRAINED TUNGSTEN INGOT WITH FUSED CENTER.  $\times 15$ .

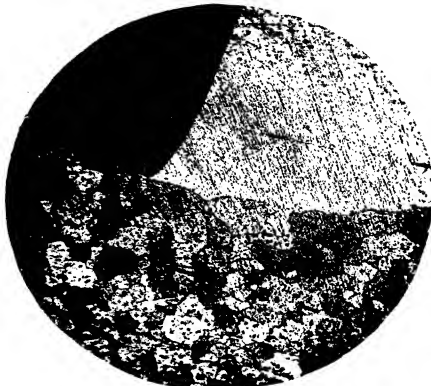


FIG. 30a.—FINE-GRAINED TUNGSTEN INGOT WITH FUSED CENTER.  $\times 144$ .



FIG. 31.—COARSE-GRAINED TUNGSTEN INGOT WITH FUSED CENTER.  $\times 17$ .



FIG. 32.—INGOT WITH TUNGSTEN-CARBIDE NETWORK SURROUNDING TUNGSTEN GRAINS.  $\times 950$ .

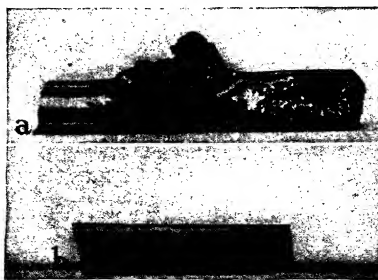


FIG. 33.—a. TUNGSTEN INGOT CONTAINING BOTH  $\text{ThO}_2$  AND CARBON. b. TUNGSTEN INGOT CONTAINING ONLY  $\text{ThO}_2$ .



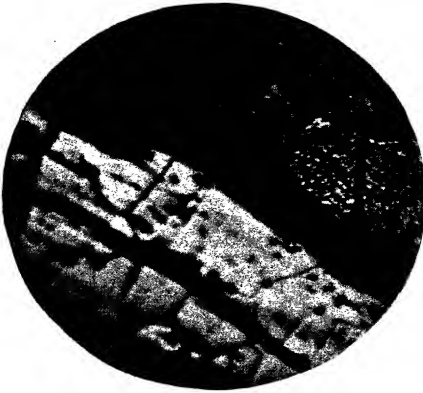


FIG. 34.—TUNGSTEN ROD CARBONIZED WITH CO. LIGHT PORTION IS TUNGSTEN CARBIDE.  $\times 940$ .



FIG. 35.—TUNGSTEN ROD CARBONIZED WITH HYDROCARBONS. LIGHT PORTION IS TUNGSTEN CARBIDE.  $\times 400$ .

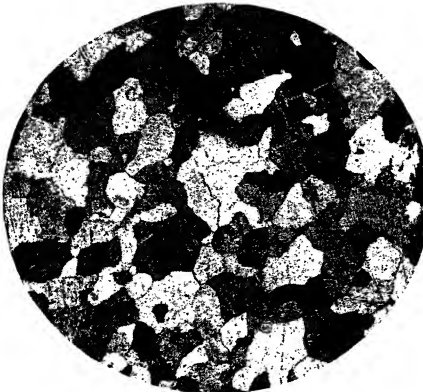


FIG. 36.—FINE-GRAINED TUNGSTEN INGOT, SWAGED AND RECRYSTALLIZED.  $\times 74$ .



FIG. 37.—COARSE-GRAINED TUNGSTEN INGOT, SWAGED AND RECRYSTALLIZED.  $\times 74$ .

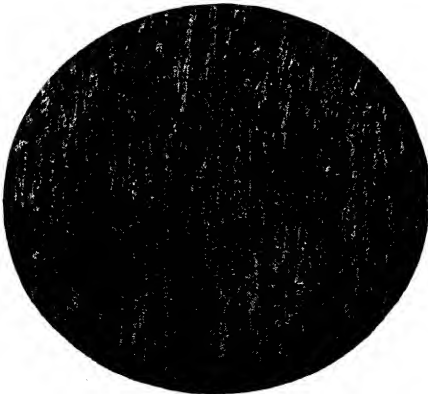


FIG. 38.—STRUCTURE OF SWAGED TUNGSTEN ROD, 0.80 IN. DIAM.  $\times 240$ .



FIG. 39.—SWAGED TUNGSTEN ROD CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ , 0.080 IN. DIAM. RECRYSTALLIZED BY HEATING 3 MIN. AT  $3100^\circ \text{C}$ .  $\times 240$ .





FIG. 40.—SWAGED TUNGSTEN ROD CONTAINING NO  $\text{ThO}_2$ . 0.080 IN. DIAM. HEATED BY ELECTRIC CURRENT SLOWLY TO  $3100^\circ\text{C}$ .  $\times 73$ .



FIG. 41.—SWAGED TUNGSTEN ROD, 0.080 IN. DIAM., CONTAINING NO  $\text{ThO}_2$ . HEATED BY ELECTRIC CURRENT QUICKLY TO  $3100^\circ\text{C}$ .  $\times 73$ .

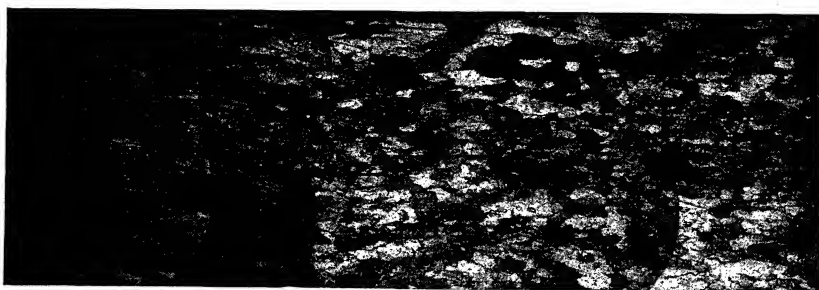


FIG. 42.—SWAGED TUNGSTEN ROD, 0.080 IN. DIAM., CONTAINING NO  $\text{ThO}_2$ . HEATED BY ELECTRIC CURRENT 2 HR. AT  $3000^\circ\text{C}$ . END NEAR COLD ELECTRODE CLAMP.  $\times 33$ .



FIG. 43.—SWAGED TUNGSTEN ROD, 0.080 IN. DIAM., CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ . HEATED  $9\frac{3}{4}$  HR. AT  $3000^\circ\text{C}$ . COLD END NEAR ELECTRODE CLAMP.  $\times 155$ .



FIG. 44.—SAME ROD AS FIG. 43, IN HOTTER REGION.  $\times 155$ .



Fig. 49.—TUNGSTEN FILAMENT 0.0015 IN. DIAM., CONTAINING 0.75 PER CENT.  $\text{ThO}_2$  AFTER BURN-OUT IN 40-WATT VACUUM LAMP. LONGITUDINAL SECTION.  $\times 346$ .



Fig. 45.—TUNGSTEN WIRE, 0.003 IN. DIAM., CONTAINING NO  $\text{ThO}_2$ . HEATED BY ELECTRIC CURRENT QUICKLY ABOVE GERMINATIVE TEMPERATURE.  $\times 355$ .

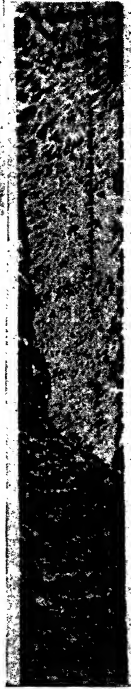


Fig. 46.—SAME WIRE AS FIG. 45. HEATED BY ELECTRIC CURRENT SLOWLY THROUGH GERMINATIVE TEMPERATURE RANGE.  $\times 355$ .



Fig. 48.—TUNGSTEN WIRE, 0.007 IN. DIAM., CONTAINING 0.75 PER CENT.  $\text{ThO}_2$ . RECRYSTALLIZED BY SHORT HEATING AT ABOUT  $2000^\circ \text{C}$ .  $\times 130$ .

Fig. 47 ON NEXT PAGE.

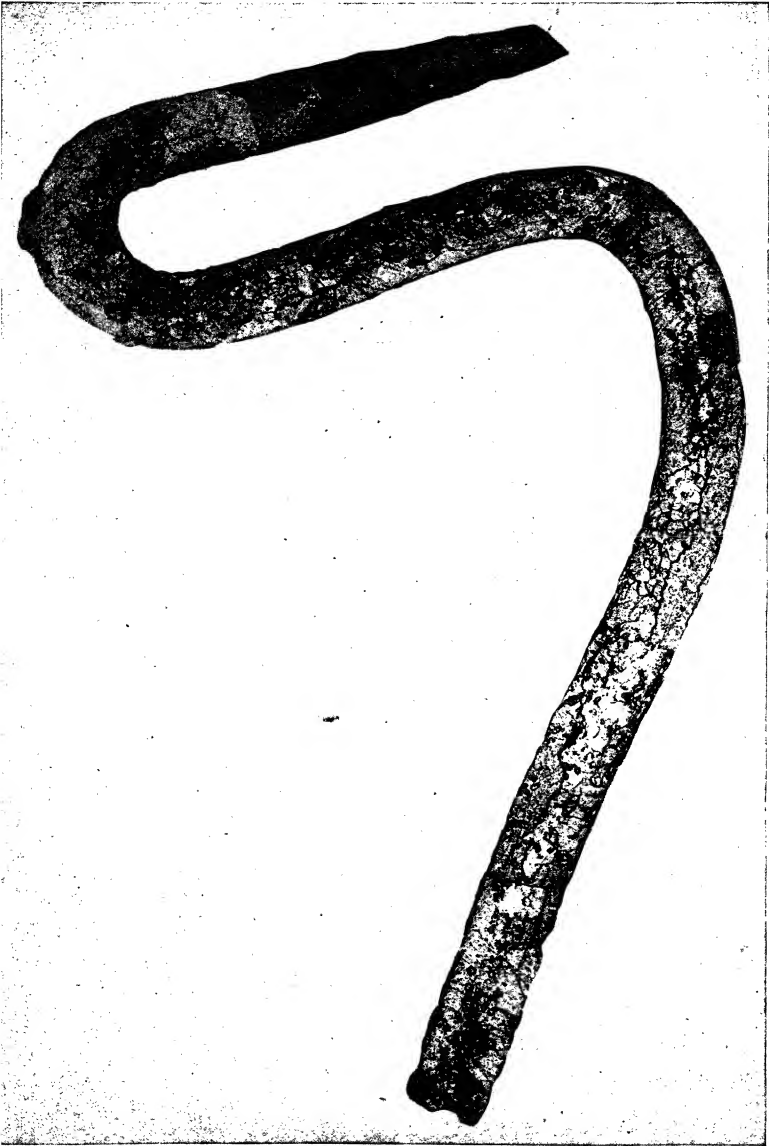


FIG. 47.—MOLYBDENUM HOOK USED AS FILAMENT SUPPORT IN 1000-WATT GAS-FILLED LAMP. LONGITUDINAL SECTION.  $\times 104$ .

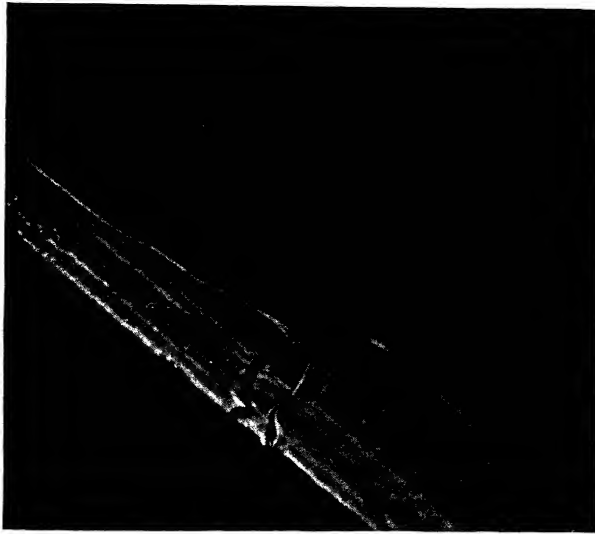


Fig. 52.

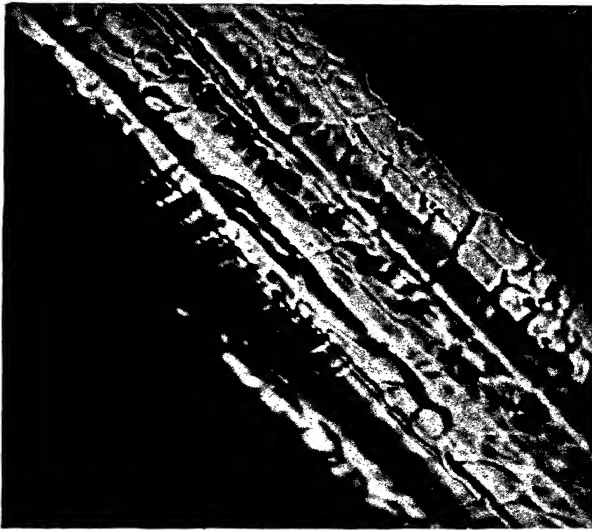


Fig. 51.



Fig. 50.

- FIG. 50.—OLD TYPE "SQUIRTED" TUNGSTEN FILAMENT AFTER BURN-OUT IN VACUUM LAMP. (YOUNG.)  $\times 325$ .  
 FIG. 51.—TUNGSTEN WIRE, 0.0036 IN. DIAM., CONTAINING 0.75 PER CENT.  $\text{ThO}_2$  AFTER 10 MIN. EXPOSURE AT  $2900^\circ\text{C}$ . NATURAL SURFACE.  $\times 480$ .  
 FIG. 52.—TUNGSTEN WIRE, 0.0036 IN. DIAM., CONTAINING NO  $\text{ThO}_2$  AFTER 10 MIN. EXPOSURE AT  $2900^\circ\text{C}$ . NATURAL SURFACE.  $\times 480$ .



Fig. 53.—TUNGSTEN WIRE, 0.007 IN. DIAM., CONTAINING NO  $\text{ThO}_2$ . USED AS FILAMENT IN VACUUM LAMP, AFTER 66 HR. BURNING. SHOWS BOTH LARGE GRAINS AND ABSENCE OF GRAIN BOUNDARIES EXTENDING ACROSS WHOLE SECTION OF WIRE.  $\times 60$ .



Fig. 54.—SPLIT TUNGSTEN WIRE CAUSED BY "OVERWORKING" OR WORKING TOO COLD.  $\times 170$ .

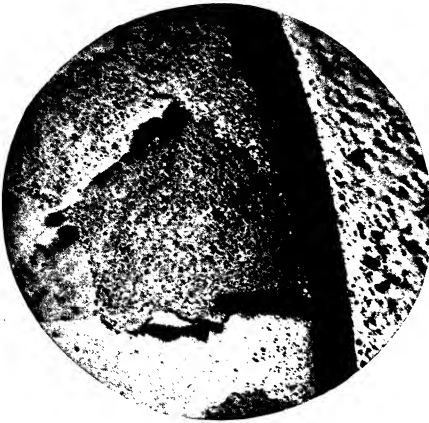


FIG. 55.—CRACKS AT GRAIN BOUNDARIES  
IN TUNGSTEN INGOT.  $\times 17$ .



FIG. 56.—CRACK PARTLY THROUGH GRAIN  
IN TUNGSTEN INGOT.  $\times 50$ .



FIG. 57.—CRACK BETWEEN TWO  
GRAINS IN TUNGSTEN INGOT. STOPS AT  
JUNCTION BETWEEN COARSE- AND FINE-  
GRAINED REGIONS.  $\times 61$ .



FIG. 58.—SHOWS CRACK ALONG  
GRAIN BOUNDARIES IN FINE-GRAINED  
TUNGSTEN INGOT.  $\times 144$ .

## DISCUSSION

SIR ROBERT HADFIELD, London, England (written discussion\*).—We have in the past known so little about tungsten that an important paper such as Mr. Zay Jeffries contributes is most welcome.

I have not much personal knowledge on the subject except as regards combinations of iron with tungsten. I put forward the results of my research on natural alloys in 1903,<sup>1</sup> and so far as I know the facts there presented still stand.

Singular to say, tungsten added to iron affects the electrical resistance less than any other added element. The following tables taken from research work by myself and Sir William Barrett may be of interest.

TABLE 1.—*Approximate Specific Electrical Resistance of Iron Alloys at Different Percentages of the Added Element*

Alloy of Iron with	Percentage of Added Element				
	1	2	3	4	6
Tungsten.....	15.5	16.5	17.2	18.0	18.5
Nickel.....	19.0	21.0	23.0	25.0	27.0
Chromium.....	....	....	24.0	26.5	29.0
Manganese.....	23.5	28.0	31.0	34.0	39.0
Silicon.....	....	....	46.0	53.5	69.0
Aluminium.....	27.0	38.0	48.0	57.0	74.0

TABLE 2.—*Increase in Specific Electrical Resistance (Microhms per Cubic Centimeter at 18° C.) Produced in Iron of Corresponding Purity by Alloying It with Various Percentages of the Elements Named*

Alloy of Iron with	2 Per Cent.	4 Per Cent.	6 Per Cent.
Tungsten.....	4.0	5.5	6.0
Nickel.....	6.5	10.0	12.5
Chromium.....	....	12.0	14.0
Manganese.....	15.0	21.0	26.0
Silicon.....	....	41.0	55.5
Aluminium.....	25.5	44.5	61.5

The following is the summing up of our results: Selecting the middle column of figures and dividing it by 4, we get the increase of re-

\* Received Aug. 13, 1918.

<sup>1</sup> *Journal, Iron and Steel Institute* (No. II, 1903) 64, 14.

sistance produced by the addition of 1 per cent. of the element in an alloy containing about 4 per cent. of the added element. This is shown in the next table, and side by side are shown the specific heat and the atomic weight of the elements named in the first column.

TABLE 3

Iron Alloyed with	Specific Resistance of 1 Per Cent.	Specific Heat	Atomic Weight
Tungsten.. . . . .	1.1	0.035	184
Cobalt.... . . . .	2.2	0.107	59
Nickel..... . . . .	2.5	0.109	59
Chromium. . . . .	3.0	0.1(?)	52
Manganese. . . . .	5.2	0.122	55
Silicon.... . . . .	10.3	0.183	28
Aluminium.. . . .	11.1	0.212	27

PAUL D. MERICA,\* Washington, D. C. (written discussion†).—This paper is a discussion of some of the results of a recent investigation<sup>2</sup> of Prof. Zay Jeffries, and of his interpretation and generalizations from these results. This work I have followed with great interest and with appreciation of its value in clarifying our views on the nature of deformation in metals and its relation to structure. It is only by such close study of the relation of structure to mechanical properties that we shall ever be able to describe the latter in terms of their ultimate elements, and thus explain what are still mysteries in the mechanical behavior of metals; experiment and thought along these lines are in my opinion of the utmost value. If I, therefore, here record some of the difficulties I have experienced in understanding some of the generalizations of Prof. Jeffries, it is only in order that perhaps from the discussion may emerge a clearer and more consistent statement of hypothesis or theory in explanation of the facts discovered by him.

Before considering the author's propositions in detail, I wish to group a few concepts and assumptions with which I here have to deal.

(1) The amorphous-metal hypothesis postulates crystalline and amorphous metal; the former, the substance of the grain in crystal, the

\* Metallurgist, U. S. Bureau of Standards.

† Received Sept. 28, 1918.

<sup>2</sup> Article 1: The Metallography of Tungsten, the article which precedes this discussion.

Article 2: The Amorphous Metal Hypothesis and Equi-cohesive Temperatures. *Journal, American Institute of Metals* (1917) 11, 300.



latter existing (a) as a cement between grains, (b) in metal deformed below the recrystallizing temperature range, at the planes along which slip has occurred.

(2) The amorphous metal at the grain boundaries and that formed at a slip plane may or may not be identical in properties, but I shall make

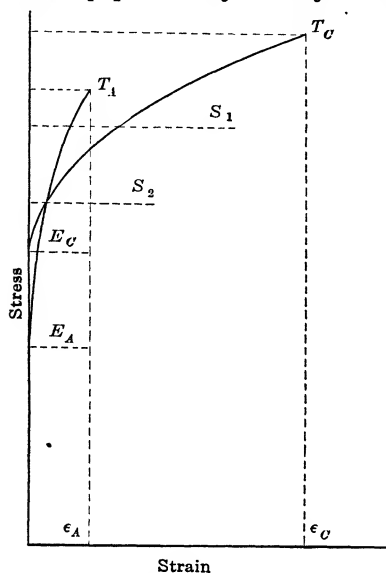


FIG. 1.—TENSILE STRESS-STRAIN CURVES FOR AMORPHOUS (A) AND CRYSTALLINE (C) METAL. ELONGATIONS PLOTTED ARE PERMANENT ONES.

that as an initial assumption; this assumption is tacitly made by the author.

(3) Each variety of metal, crystalline (C) and amorphous (A), has its own mechanical characteristics, with three of which I shall be concerned: the ultimate tensile strength, the elastic limit, and the elongation at rupture. Neglecting the phenomenon of "necking-down" at rupture, which is a consequence of non-uniformity either of stress or of strength along the axis of a bar in tension, the stress-strain curves of C and of A metal may be assumed to have the general form shown in Fig. 1. The C metal has a definite elastic limit, and its properties are directional. The A metal probably has no definite elastic limit, but we may assume a value  $E_A$  at which the permanent deformation exceeds a certain arbitrary value; the properties of A are not directional, but depend undoubtedly upon the rate or duration of application of stress; *i.e.*, the A metal is viscous in its nature.

(3a) It is likely that the elastic limit of a grain can be increased in any direction, as Tammann has shown, by the gradual exhaustion of the most accessible slip planes, but the ultimate tensile strength of the crystalline metal of a grain in any direction probably is a function only of temperature. The curves of  $E_C$  shown in Fig. 2 are to be considered as those of the undeformed metal; the values may be raised possibly up to those of  $T_C$ .

(4) The effect of temperature on the shape of these curves is to diminish  $T_A$ ,  $T_C$ ,  $E_A$ , and  $E_C$ , and to increase  $\epsilon_A$  and  $\epsilon_C$ .

(5) The tensile ductility is measured by the amount of permanent set per unit length occurring between  $T$  and  $E$ ; it is undoubtedly a function of the rate of loading between these two values of stress. Exhaustion of ductility occurs, as Prof. Jeffries points out, not because the metal cannot be further plastically deformed, but because the tensile load necessary to produce the deformation will first rupture the bar. According

to Prof. Jeffries, the variations in the values of  $E_A$  and  $E_C$ , respectively, with temperature are such that for every metal (and alloy) there is a temperature ( $EC_E$ ) below the melting point, such that

$$\begin{array}{ll} \text{above } EC_E & E_C > E_A \\ \text{at } EC_E & E_C = E_A \\ \text{below } EC_E & E_C < E_A \end{array}$$

This is the equi-cohesive temperature; see Fig. 2, in which  $E_A$  and  $E_C$  are shown as functions of temperature.

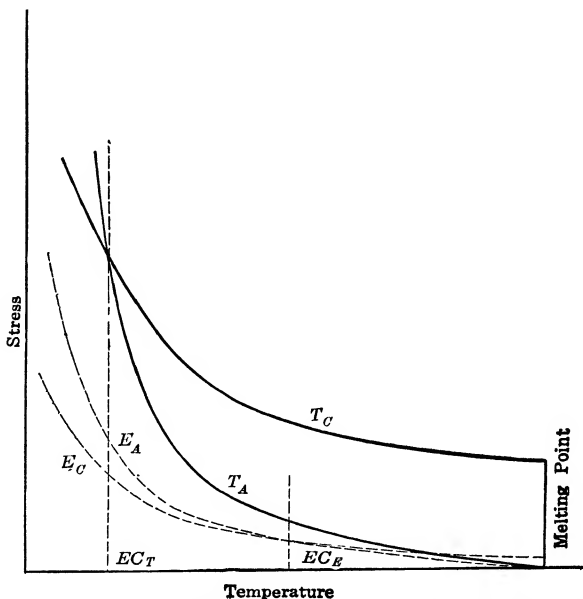


FIG. 2.—EQUI-COHESIVE TEMPERATURES AND EFFECT OF TEMPERATURE ON  $E$  (UNDEFORMED) AND  $T$ .

(6) I should like to call attention to another equi-cohesive temperature, namely, that at which the tensile strength of  $A$  and  $C$  metal are equal; such a temperature has been predicted by Rosenhain.<sup>3</sup> This is shown in Fig. 2;  $EC_E$ , the equi-cohesive temperature for elastic limits, and  $EC_T$ , that for tensile strength, are not necessarily equal.

(7) When a metal ruptures in tension, the path of rupture is never straight; it passes sometimes through the grains (intracrystalline) and sometimes along the grain boundaries (intercrystalline). The rupture along those portions not perpendicular to the direction of the net tension occurs, very probably, partly as a result of shearing stresses. The load necessary to cause fracture is probably proportional to the area of the path of rupture.

<sup>3</sup> Walter Rosenhain: "An Introduction to the Study of Physical Metallurgy." New York, 1914. D. Van Nostrand Co.

(8) In any case, I wish to make two assumptions regarding rupture:

(a) That the total force to cause rupture is proportional directly to area of its path, counting sheared areas as equal in effect to those separated in direct tension.

(b) That the path of rupture across a grain, which usually appears to be straight, but which may involve serrations such as to increase its apparent length, is actually no longer than the path around the boundary of a spherical grain of which the apparent path is a diametral plane.

(9) In case the grain is elongated perpendicular to the general direction of stress and path of rupture, there are three potential paths of rupture:

- (a) Across grain, through *C* metal.
- (b) Across grain, through *A* metal (at a slip plane).
- (c) Around grain, through *A* metal.

Rupture will occur along that path for which the product of area and unit strength is least.

I wish now to discuss on this basis some of the propositions suggested by Prof. Jeffries.

(A) On page 618 (Article 1), the author explains the absence of ductility at ordinary temperatures in tungsten consisting of equi-axed grains as existing "because the brittle amorphous phase at the grain boundaries permits rupture before a load sufficient to deform the malleable crystalline phase can be applied;" *i.e.*, because  $T_A < EC$  at ordinary temperatures. In his other paper (Article 2) he has found that  $EC_E$  for equi-axed tungsten was approximately 1350° C. Therefore  $E_A > EC$  below 1350° C. But, since  $T_A > E_A$ , therefore  $T_A > EC$  below 1350° C., a conclusion which is the exact opposite of the one assumption used above.

This contradiction may be explained, possibly, by the fact that the equi-cohesive temperatures which Prof. Jeffries determined<sup>4</sup> are not the equi-cohesive temperatures as he defines them on p. 311 of the same article. Reference to Fig. 1 will illustrate this; the two curves are the stress-strain curves of *A* and of *C* metal at a temperature (*t*), above the equi-cohesive temperature  $EC_E$ , as defined on p. 311 by the author; *i.e.*, such that  $E_C < E_A$  and  $t < EC_E$ . Yet at a stress,  $S_1$ , the permanent set of the crystalline metal is, however, greater than that of the amorphous metal; it will be observed that it is the temperature at which, for a given stress, the permanent deformations of *A* and *C* metal are equal, which the author determines and calls equi-cohesive temperature (p. 312).

To explain lack of ductility of equi-axed tungsten along the general line of the author's explanation, we might then assume that at ordinary temperatures we are above  $EC_E$ , not below it.

<sup>4</sup> *Journal, American Institute of Metals* (1917) 11, 312.

(B) On p. 622 (Article 1), the author says, "The underlying reason for the loss of ductility by working a metal at a certain temperature below its annealing temperature" ( $EC$  probably) "and the regaining of ductility by cooling to some lower temperature, is that the amorphous phase of any metal will increase in cohesion on cooling, at a faster rate than the crystalline phase." I believe that this is not quite accurately stated; the necessary conditions to produce this effect are not so simple. They appear to me to be as follows:

(1) Since the strength of the  $A$  metal is to determine the ductility, fracture must take place through it; *i.e.*,

$$\phi_A T_A < \phi_C T_C$$

where  $\phi_A$  and  $\phi_C$  are the areas of the potential paths of rupture through  $A$  and  $C$  metal, respectively.

(2) But the fact is established that fracture takes place across a deformed grain; *i.e.*, through  $A$  metal, at slip planes either in tungsten (see p. 618) or in other metals below  $EC$ .

(3) Therefore  $\phi_A = \phi_C$  (approx.) and  $T_A < T_C$ .

(4) The condition for the exhaustion of ductility is not that the "cohesion" of  $A$  and of  $C$  become equal, but that  $I_A$  is not equal to nor greater than  $E_C$ .

(5) In order that the ductility of deformed metal may reappear upon lowering the temperature, the quantity  $(T_A - E_C)$  must increase with this lowering.

These conditions are represented in Fig. 2, and are quite possible ones; in fact the author's hypothesis could quite well fix the case of tungsten. But it is to be observed that condition (3) would require that in any metal within the temperature range within which ductility after deformation was restored upon lowering the temperature, fracture should occur within the same temperature range in an equi-axed grain at the grain boundaries: *i.e.*, this range is above  $EC_T$ .

But this is not true of gold, silver and copper, in which this identical phenomenon occurs. In these metals, at those temperatures,  $T_A > T_C$ , and the amorphous metal does not determine strength nor ductility.

(C) There is, however, a further contradiction in the author's views to which I wish to call attention. On p. 618 (Article 1) he says that fibrous tungsten "is ductile because the grain distortion by working arranges the grain boundaries in such a manner that the resistance to rupture along them is so great that rupture is forced to take place through the deformed grains themselves." In other words, equi-axed tungsten is brittle because  $\phi_A T_A < \phi_C E_C$ , where  $\phi_A = \phi_C$  approx. When the grains are elongated,  $\phi_A$  becomes large enough that  $\phi_A T_A > \phi_C E_C$ , and the fracture takes place across the grain. Now, on the basis of assumption (2) above, this fracture cannot be through  $A$  metal; for we should have,

in that case,  $\phi_{A_1}T_A$  (of fibrous grains)  $> \phi_{A_2}T_A$  (of equi-axed grain). But  $\phi_{A_1} = \phi_{A_2}$  (approx.) if the path of rupture is across grain; therefore  $T_A > T_A$ , an obvious absurdity. Therefore, axial fracture in fibrous tungsten must be through the crystalline metal; *i.e.*,  $T_A > T_c$ . But this contradicts the assumption (1) under (B), necessary to explain the regaining of ductility of deformed tungsten upon lowering the temperature.

These are some of the difficulties I have experienced in reconciling the various views of Prof. Jeffries, which lead me to wonder whether we must not examine more closely into the tacit assumptions which are made in developing such hypotheses, and also into the exact nature of deformation, before we may attempt to generalize to any too great extent. Such questions as the following must receive more thorough attention:

(1) Is amorphous metal the same in properties whether at slip planes or at grain boundaries?

(2) What is the actual shape and area of a fracture across a grain; in other words how serrated is this path?

(3) Does fracture across a grain occur within crystalline or within amorphous metal?

(4) What is the nature of the resistance to plastic deformation within a grain? This resistance would seem to be in reality a very complex function.

I believe the recent experimental results of Prof. Jeffries, as well as those of other investigators whom he has quoted, can contribute much to the elucidation of these specific questions.

J. C. W. HUMFREY,\* Sheffield, England (written discussion†).—Prof. Jeffries' paper gives a very complete description of the metallurgy of tungsten, the details of which afford a striking illustration of the application of scientific principles in overcoming practical difficulties of manufacture. In addition to this practical side, it deals with two phenomena of great theoretical importance and of general metallographic interest, *viz.*:

(a) Grain growth in metals.

(b) The significance of intercrystalline cohesion.

It is chiefly with the second of these two subjects that I propose to deal in the following remarks.

On page 617 the author states that he accepts the amorphous boundary theory, *viz.*, that the crystals in a mass of metal are joined to one another by a cement of the same metal, in the amorphous or non-crystalline state. Agreeing, as I also do, with the validity of this theory, I am unable to accept certain of the interpretations he puts upon it, or to follow his arguments as to the manner in which his experimental data may be taken as a confirmation of its truth.

\* Admiralty Inspection Officer.

† Received Sept. 28, 1918.

The author appears to consider the amorphous cement as occupying a volume in the mass comparable with that of the crystals, and deals with the mechanical properties of the mass as if the two phases were distinct from one another and possessed little or no mutual adhesion. His comparison of the structure of tungsten with a mass of iron crystals embedded in glass is not, I think, a very happy one, nor is it indicated either by the mechanical properties of metals in general nor of this one in particular.

In a paper (which he quotes in the bibliography, No. 33) I put forward a theory based on the view that the amorphous cement owes its origin to the impossibility of two differently oriented crystals fitting exactly into one another, and the consequent existence of a certain range of atoms between them which, since they are being constrained by each crystal in a different manner, are forced to take up some irregular grouping between the two. According to this theory, there is no sharp line of demarcation between crystalline and amorphous, and the one must merge gradually into the other. The whole thickness of the disturbed layer is of molecular rather than of microscopic dimensions, and, while the amorphous envelopes are incapable of plastic deformation, yet they are capable, in consequence of their extreme thinness, of considerable distortion relative to the crystals they surround by means of elastic bending.

In all metals which are at temperatures considerably below that of their melting points, the amorphous phase is, as is illustrated in the author's Plate 3 (page 623), of such an extremely viscous nature as to be incapable of plastic deformation, but to possess very considerable tenacity or cohesion. If, therefore, we accept the author's theory that the intercrystalline fracture of his tungsten is due to the brittleness of the amorphous cement, then such a form of fracture should be the rule rather than the exception in other metals. The paper refers to one other case only, viz., iron at the temperature of liquid air; but the author does not state whether he has obtained direct experimental confirmation of the fact, nor does he quote any authority to support it. In Hadfield's paper (No. 38 in the bibliography), while it was shown that iron was brittle at this low temperature, yet I can find no reference as to whether the brittle fractures followed the crystal boundaries or the crystal cleavages. Since, however, the brittleness was accompanied by a high tenacity, I should prefer to assume the latter rather than the former.

When a metal tends to fracture around rather than through the crystals, it must surely be an indication that the *tenacity* of the cement is less than that of the crystals, and, provided the thickness of the cement is sufficiently small, its lack of plastic ductility does enter into the matter. All metals in their normal state tend to fracture around the crystals when at temperatures just below those at which they freeze (bibliography, No. 31). In this range, the amorphous cement is more capable

of plastic deformation than the crystalline, but possesses considerably less tenacity. Below the temperatures which the author terms those of "equi-cohesion," the fractures change to cleavage. If, at still lower temperatures, the fractures again reverted to intercrystalline, then it would have to be assumed that the tenacity of the amorphous phase had begun to decrease with temperature, or at least to increase at a lower rate than the crystalline, and that the two curves in the author's Plate 3 again crossed one another. Such a réversion is most unlikely, and opposed to all our experimental knowledge.

We do, however, know of cases in which certain metals, which normally break through the crystals, may be so altered in character as to tend to break around them. Typical examples of this are:

(a) Gold containing small percentages of bismuth. It has been shown that the weakness is due to the presence of a fragile gold-bismuth eutectic.<sup>5</sup>

(b) Pure iron which has been annealed at certain temperatures and in certain atmospheres, and slowly cooled through a certain range. In this case there is evidence to indicate that the weakness is due to the formation of an iron-oxide eutectoid.<sup>6</sup>

(c) Nickel-chrome and certain other alloy steels, when hardened by quenching, annealed below the critical range, and then allowed slowly to cool through a range of temperature in the neighborhood of 500° C. In this case there is as yet no experimental evidence to indicate what is the important factor, but the general nature of the phenomena again points to the formation of a eutectoid.<sup>7</sup>

In the above examples it would appear that, contrary to the normal amorphous cement being responsible for the intercrystalline weakness, it is rather the fact that the presence of some foreign material between the crystals has interfered with its proper formation. It is suggested that it is to some similar interfering agent that the weakness found in tungsten, as made by the process now employed, should more properly be ascribed. Future researches will, I think, find a method of preparing metallic tungsten which shall be free from such weakness and have mechanical properties in line with those of other metals. The preparation of such material would be of great practical importance and would well repay the labor of a lengthy investigation.

It is fairly obvious that severe deformation by such processes as wire

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<sup>5</sup> J. O. Arnold and J. Jefferson: Influence of Small Quantities of Impurities on Gold and Copper. *Engineering* (Feb. 7, 1896) **61**, 176-179.

<sup>6</sup> J. C. W. Humfrey: The Intercrystalline Fracture of Iron and Steel. *Carnegie Scholarship Memoirs*, Iron and Steel Institute (1912) **4**, 80-105.

<sup>7</sup> H. P. Philpott: Some Experiments on Notched Bars. March, 1918, meeting, Institute of Automobile Engrs. Will probably be published in *Proceedings* (1918-19) **12**.

drawing or swaging will tend to strengthen a metal which originally lacks intercrystalline cohesion, since it must result in a certain amount of dovetailing of the crystals into one another. If, however, it is assumed that the original brittleness was due to the presence of the amorphous cement, it would appear that the contrary would be the case, since many additional planes of weakness would be produced by the formation of amorphous matter in the slip planes.

Cold working of a metal must necessarily, I think, be accompanied by the formation of internal stresses, partly due to unequal external loading, partly to the difference in resistance offered by the crystals and the amorphous matter surrounding them and formed during deformation, and partly to varying plasticities of different crystals, owing to the way they are oriented to the main axes of strain. When a certain degree of deformation has been given, the internal stresses at certain places may rise to that of the maximum tenacity of the metal. Further deformation at the same temperature must then immediately lead to internal rupture; this is the state of maximum tenacity which can be produced in the metal by cold work. If, however, the temperature of the metal be reduced, the tenacities of both the crystalline and the amorphous phases are increased, and further deformation may now be applied before the internal stresses again become equal to the tenacities, *i.e.*, the metal has acquired additional ductility. This explanation is applicable to all metals, including tungsten, and is independent of the relative tenacities of the crystalline and amorphous phases, provided only that both increase with falling temperature.

ZAY JEFFRIES (author's reply to discussion\*).—Messrs. Merica's and Humfrey's criticisms of some of my conclusions are rendered in the true scientific spirit by means of which differences of opinion are gradually smoothed out and all participants are benefited. It is true that the statement of generalizations on such a complex subject is hazardous, but I shall endeavor to point out the explanations for the discrepancies noted. Owing to the fact that I am now submitting a long paper on the same general subject to the Institute, my present remarks will be brief.

*Dr. Merica's Assumptions*

I agree with No. 1 and No. 2, with the modification that the A (amorphous) metal between grains of annealed metal has substantially the same properties as that at slip planes in deformed metal, but the two have different arrangements with reference to C (crystalline) metal and they may be in different conditions as regards internal stress, thickness, and nature of contact with C metal.

Assumption 3.—I believe Fig. 1 is wrong. I know of no evidence to

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\* Received Oct. 30, 1918.



substantiate the curves. I believe  $\epsilon_c$  (elongation of  $C$  phase) = 0 (approximately) at all temperatures and  $\epsilon_A$  (elongation of  $A$  phase) has finite values at elevated temperatures only; but these play no important direct role in the ordinary deformation of metals at low temperatures. I believe  $E_c$  (elastic limit of  $C$  phase) =  $T_c$  (tensile strength of  $C$  phase) because as soon as  $C$  metal begins to deform permanently it would rupture were it not for the  $A$  metal generated by its breakdown. We know that  $E_c = T_c$  in brittle crystals and a crystal cannot be permanently deformed without generation of  $A$  metal. Therefore Dr. Merica's  $T_c$ , in reality, equals  $T_{(A+c)}$ . Also  $E_A = T_A$  for all practical purposes, because when  $A$  metal permanently deforms no new stronger phase is formed and hence any unit load which will permanently deform it will eventually break it. If the above is true, Dr. Merica's Fig. 2 is wrong, there being but one set of curves for  $E_A$  and  $E_c$  which correspond to my Plate 3. The duration of load is very important in  $A$  metal. The above reasoning should also be applied to assumptions 4 and 6.

Assumption 8b.—I believe this is not warranted from the evidence at hand and may lead to wrong conclusions.

Assumption 9.—I should like to add a fourth potential path of rupture, namely, at the contact between  $A$  and  $C$  metals.

#### *Dr. Merica's Conclusions*

(a) I have concluded, after examining considerable evidence, that at room temperature and lower, in equiaxed tungsten, there is internal stress between  $A$  and  $C$  metals, probably due to differences in their coefficients of thermal expansion. Call this internal stress  $IS$  and Dr. Merica's  $T_A < E_c$  becomes  $T_A$ , or  $E_A - IS < E_c$ . My  $EC_E$  (equicohesive temperature for elastic limit) is not as Dr. Merica states; it is the temperature at which mixtures of  $A$  and  $C$  metals have the same resistance to deformation regardless of the initial quantity of  $A$  metal present. Obviously, when  $C$  metal or a mixture of  $A$  and  $C$  metals is deformed by Brinell impression,  $A$  metal is generated. If the total load is kept constant, the deformation gradually increases the area of contact of the ball and decreases the unit load until a point is reached at which substantially no further deformation takes place; this measures the resistance to deformation of mixtures of  $A$  and  $C$  metals. The same is true of my hairpin tests; the deformation caused a lessening of the load by shortening the lever arm. In all cases the deformation was caused, to my mind, largely by the movement at the slip planes and slightly, and more particularly at the higher temperatures, by actual deformation of  $A$  metal.

(b) Dr. Merica's condition for the increase in ductility at lowered temperatures is that the quantity  $(T_A - E_c)$  must increase with decrease in temperature. In view of the assumption that  $T_A = E_A$ , Dr. Merica's

condition is identical with mine. It seems to me that increase in ductility of a deformed metal by lowering the temperature has no relation to rupture at grain boundaries; the former holds for the range from highest temperature down to that of maximum ductility and the latter takes place only when the internal stresses between *A* and *C* metals exceed a certain amount. Briefly, the cohesion of *A* and *C* metals, the quantity and arrangement of the *A* metal and time of rupture (whether simultaneous or gradual) and rate of loading (especially at high temperatures) will determine the breaking load at a given temperature in the ductile temperature range. With other conditions constant, the breaking load will increase with decrease in temperature proportional to the cohesion of *A* metal. But with decrease in temperature the elastic limit of the deformed grains, which consist of *A* and *C* metals, must increase less than the elastic limit of *A* metal and more than the elastic limit of *C* metal. Hence the breaking load increases faster than the elastic limit of deformed grains and ductility must increase with decrease in temperature.

(c) Again Dr. Merica's discrepancy is explained by the existence of internal stress between *A* and *C* metals. It seems to me that Dr. Merica has unnecessarily complicated this subject by the assumption that the tensile strengths and elastic limits are not equal. With my assumptions,  $E_A$  is the average elastic limit of *C* metal in all directions and  $E_A$  is greatly affected by the duration of the load, Dr. Merica's Fig. 2 assumes that  $T_A = E_A$  at the melting point. We are all familiar with the brittleness of amorphous substances at very low temperatures, which means that at these temperatures  $T_A = E_A$  also. According to Fig. 2,  $E_A$  and  $T_A$  become farther apart as the temperature decreases and hence could never intersect. Also, Dr. Merica's  $T_C$  and  $E_C$  curves cannot intersect at low temperatures, yet we know at these temperatures they are equal. The obvious conclusion is that  $T_A = E_A$  and  $T_C = E_C$ .

#### *Reply to Mr. Humfrey*

Mr. Humfrey first gives his conception of the quantity and arrangement of the *A* metal and states that I consider the amorphous cement as occupying a volume in the mass comparable with that of the crystals. My opinions on this subject coincide in the main with Mr. Humfrey's, and I have so stated in reference 34.

My previous remarks on internal stresses between *A* and *C* metals at low temperature apply to Mr. Humfrey's suggestions as to the nature of intercrystalline cohesion. He makes one statement, however, which, on its face, seems to be a contradiction to the whole hypothesis; namely, that if the *A* metal at the grain boundaries gives way for any cause at a load lower than the elastic limit of the *C* metal, the *A* metal at the slip planes in the deformed grains should do the same. It is a fact—and this will be discussed fully in my next paper—that the *A* metal at the

slip planes does produce brittleness and, eventually, weakness in the deformed grains, but the effect of the deformation is to lower the temperature at which this brittleness and weakness first appear on cooling. The intercrystalline fracture occurs in recrystallized tungsten at 200° C. and lower. Below room temperature, equiaxed tungsten becomes weaker with decreasing temperature; also, severely worked tungsten is weaker in liquid air than it is at room temperature, and it is also brittle. The above indicates that either the path of rupture along the amorphous slip planes in deformed grains is greater than at grain boundaries in equiaxed tungsten, or that the *A* metal in deformed grains which has been generated at a comparatively low temperature must be cooled to a lower temperature than that at the grain boundaries to produce a given amount of internal stress between *A* and *C* metals.

Mr. Humfrey's belief that future researches will find a method of preparing metallic tungsten which will be free from intercrystalline weakness has been shared by many workers on tungsten. Although recognizing that such a thing may be possible, I am of the opinion that fine-grained equiaxed tungsten will never be produced in such a form that it will be ductile at ordinary temperatures like other metals. Lamp filaments recrystallized in a vacuum and heated at high temperatures for over 1000 hr. are not ductile at ordinary temperature unless a single grain occupies the whole cross-section of the filament for a considerable length, or the grains are greatly elongated, as indicated in Fig. 49. Tungsten rods composed of large grains can be worked and recrystallized in such a manner that each large grain gives birth to many small equiaxed grains which show intercrystalline fracture at room temperature. If we assume that the original large grain should have concentrated its impurities at its own boundary, the small grains produced from it should be cemented together with substantially pure *A* metal. Again, the *A* metal at the slip planes in deformed tungsten grains behaves like the intercrystalline material when a sufficiently low temperature is reached.

I am unable to follow Mr. Humfrey's reasoning with reference to the increase in ductility in a strain-hardened metal with decrease in temperature. It is true that an increased tenacity due to release of strains increases ductility at a given temperature, but when the increased tenacity is gained by lowering the temperature, it must be remembered that the resistance to deformation increases also. What makes the tenacity increase faster than the resistance to deformation, Mr. Humfrey's explanation does not say. My contention that in ductile metals the amorphous metal controls the tenacity and that its cohesion increases with decrease in temperature faster than the crystalline metal, satisfies the above conditions, it seems to me, and furthermore, this generality holds for all metals.

## Metallographic Phenomena Observed in Amalgams

BY ARTHUR W. GRAY,\* PH. D., MILFORD, DEL.

(Milwaukee Meeting, October, 1918)

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Phenomena which reveal chemical and physical processes that probably go on in many alloys at temperatures not far removed from their melting ranges are observable in dental amalgams. These amalgams should, therefore, be of even more interest to the metallographer, the metallurgist, the chemist, and the physicist than to the dentist who uses them for saving decaying teeth. For this reason I am here de-

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scribing a few of the results recently obtained during the course of a systematic investigation of dental alloys.<sup>1</sup>

In order to keep this account within reasonable bounds, I shall confine myself almost entirely to the consideration of strength in compression (crushing strength) and of dimensional changes during hardening (reaction expansion). Conditions influencing the mercury content<sup>2</sup> will

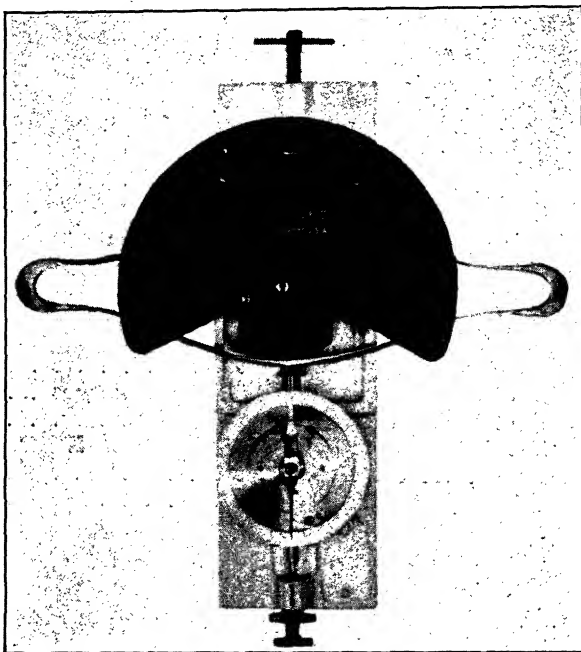


FIG. 1.—SPRING DYNAMOMETER DEVISED BY DR. BLACK FOR TESTING CRUSHING STRENGTH AND FLOW OF DENTAL FILLING MATERIALS.

receive some attention. A few results have already been communicated to the American Physical Society.<sup>3</sup>

<sup>1</sup> In this investigation I was assisted by Paris T. Carlisle, 4th, and by Miss Martha C. Borton. To both of these I am greatly indebted for the care with which they looked after the numerous experimental details and records of some thousands of tests, the routine measurements for which were made, as a rule, by young women who were trained specially for the duties assigned to them.

<sup>2</sup> This term is here used to mean the per cent. of mercury in an amalgam. See p. 662.

<sup>3</sup> A. W. Gray and P. T. Carlisle: The Influence of Temperature upon the Crushing Strength of a Dental Amalgam. Rochester Meeting. *Physical Review* (1918) 11, 194. The Influence of the Pressure and the Time Employed in Condensing a Dental Amalgam upon its Crushing Strength at Temperatures between 10° and 100° C., Pittsburgh meeting; unpublished. The Influence of Amalgamation Variables upon the Mercury Content and the Crushing Strength of a Dental Amalgam. New York meeting. *Physical Review* (1918) 11, 492.

## I. CRUSHING STRENGTH

## 1. THE BLACK DYNAMOMETER

Practically all strength tests of dental amalgams have heretofore been made with the spring dynamometer devised by Dr. Black.<sup>4</sup> The specimens tested were in the form of cubes 0.085 in. (2.16 mm.) on each edge. They were prepared by packing the amalgam into split steel molds essentially as it would be packed into a tooth cavity, sometimes with simple hand force, sometimes with blows from a mallet. The cubes were crushed at whatever temperature the room happened to offer, and the mean of several trials was taken to represent the strength (Fig. 1 and 2).

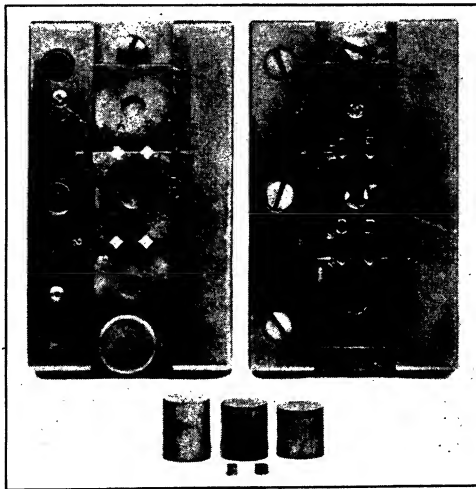


FIG. 2.—MOLDS FOR PREPARING 0.085-IN. CUBES OF AMALGAM TESTED IN BLACK DYNAMOMETER. BELOW ARE SHOWN RELATIVE SIZES OF CUBES AND CYLINDERS USED FOR CRUSHING TESTS.

Every physicist will recognize that, while such tests may be of great value in pioneer work, they are hardly likely to yield results having the accuracy desirable in modern research. The cube, with its sharp corners and its radial asymmetry, is not the best shape for a test specimen. The particular cubes used with Black's dynamometer are so small that the accidental errors arising from unavoidable irregularities in shape, size, and internal structure may easily render the results uncertain. Their smallness introduces an error owing to the restraining action of the jaws between which they are crushed. Temperature affects the stiffness of the spring used for measuring the crushing force; it also affects considerably the strength of a dental amalgam. The strength of an amalgam

<sup>4</sup> *Dental Cosmos* (1895) 37, 406 ff.

specimen is also largely dependent upon the pressure under which it is molded into form; and hand or mallet packing cannot control this pressure sufficiently well for accurate comparisons of different alloys.

#### STANDARDIZED PROCEDURE FOR CRUSHING-STRENGTH TESTS

I have, therefore, discarded the old Black dynamometer in favor of a 9000-kg. Olsen testing machine, which was designed for operation by

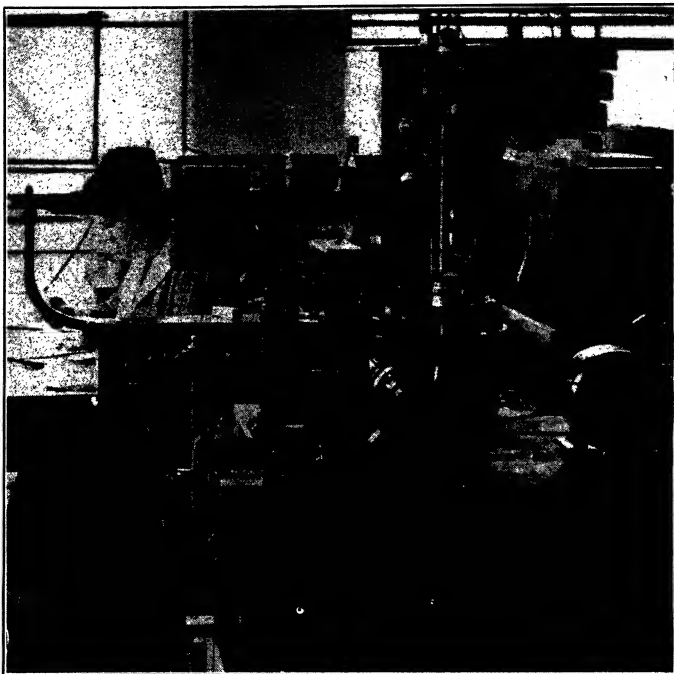


FIG. 3.—THE 9000-KG. OLSEN MACHINE ADAPTED TO TESTING STRENGTH OF MATERIALS AT ANY TEMPERATURE AND PROVIDED WITH POWER DRIVE FOR APPLYING TESTING LOAD WITH REGULARITY.

hand (Fig. 3). This I modified by the addition of an electric motor drive which applied the load with regularity, thus permitting the beam to be kept balanced to a nicety right up to the moment of failure; and by the addition of electric heaters for bringing the specimen under test to the desired temperature, which was determined by a suitably placed thermo-element. Additional thermo-elements enabled temperature gradients within the heated region surrounding the cylinder to be adjusted to negligible values. The electromotive forces were measured by a potentiometer.

The test specimens were in the form of right circular cylinders 10.04

mm. in diameter and of sufficient height to eliminate errors caused by the restraining action of the crushing jaws (Fig. 4). These cylinders were prepared by molding the freshly mixed amalgam in a thick-walled steel tube, the interior of which was highly polished. A measured force, applied by the testing machine and maintained for a measured time, was used in packing the amalgam between an accurately fitting piston and the removable bottom of the mold. This packing squeezed out the excess of mercury and condensed the plastic mass into a firm, smooth cylinder with parallel ends. The procedure secured both uniformity of cross-section and uniformity of packing conditions.

Many preliminary experiments were made to obtain a general idea of the ways in which different conditions may influence the crushing strength of a dental amalgam. These experiments showed the effect of variations in such factors as the proportions of mercury and alloy used in mixing the amalgam, the time devoted to triturating the mix, the

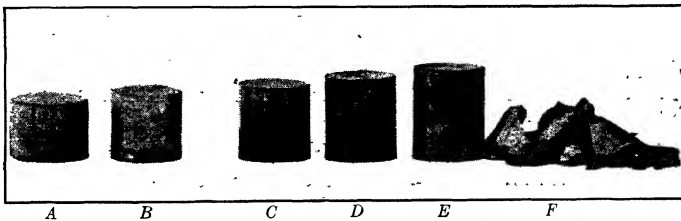


FIG. 4.—TEST CYLINDERS OF AMALGAM PREPARED BY AMALGAMATING 6 GM. ALLOY. CYLINDERS A AND B FROM LOW-SILVER ALLOY COMPRESS AND CRACK. CYLINDERS C, D, E, AND F FROM HIGH-SILVER ALLOY BURST EXPLOSIVELY. CYLINDERS B AND D, BOTH PACKED UNDER 400 KG., SHOW DIFFERENCE IN SIZE OF AMALGAM FROM SAME WEIGHT OF ALLOY. D IS 25 PER CENT. LARGER AND 75 PER CENT. STRONGER THAN B. C, D, AND E ARE PACKED UNDER 141, 400, AND 1131 KG. PER CIR. CM., RESPECTIVELY. NATURAL SIZE: 10.0 MM. IN DIAMETER.

temperature of trituration, the pressure under which the amalgam is molded into test cylinders, the time that this packing pressure is maintained, the height of the test cylinder, the time that elapses between the making and the crushing of the cylinder, the temperature at which it is stored during this interval, and the rate at which the crushing load is applied during the testing. To save time in reaching the general conclusions desired, no attempt at controlling the temperature of the specimen during crushing was made in these earlier tests; the tests were made at whatever temperature the room happened to offer. As a consequence, the accidental errors, measured by deviations from average values, were often considerably larger than in the later tests, in which temperature and other influencing conditions were carefully controlled. Even the preliminary experiments demonstrated the need of a thoroughly standardized technique if results of real value were to be obtained.



## INFLUENCE OF HEIGHT OF TEST-PIECE UPON APPARENT STRENGTH

In short-column tests it is important to have the test specimen sufficiently high to eliminate the effect of friction between the crushing jaws and the specimen; otherwise the apparent strength will be greater than the true strength of the material. Therefore, one of the first things to be determined was the minimum height allowable with amalgam cylinders 1 cm. in diameter. About 9 mm. was found to be ample; reduction to 6 mm. would not introduce serious error (Fig. 5). However, no cylinders shorter than 10 mm. were ever used.

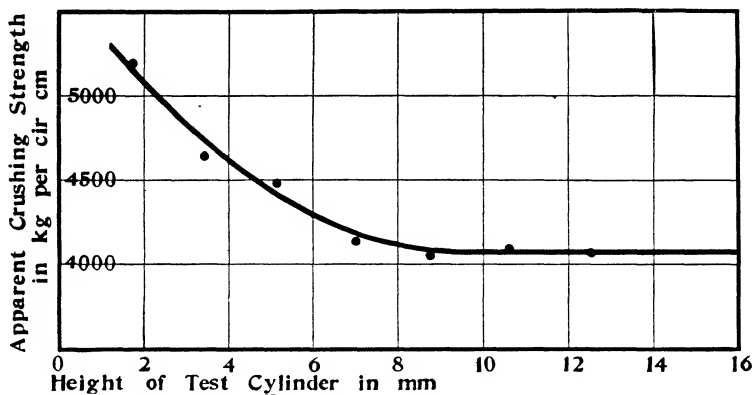


FIG. 5.—TEST SPECIMENS THAT ARE TOO SHORT MAKE MATERIAL APPEAR STRONGER THAN IT REALLY IS.

## INFLUENCE OF PACKING PRESSURE AND OF PACKING TIME

The next points to be decided were the influence of packing pressure and of packing time upon both the mercury content and the strength of a high-grade amalgam.<sup>5</sup> The pressures used in packing were systematically varied from 25 to 3200 kg. per cir. cm.;<sup>6</sup> and the time during which a packing pressure was maintained was varied from 1 to 16 minutes.

<sup>5</sup> The ratio of the masses of mercury and alloy that are mixed together to form an amalgam will be frequently referred to as the "mercury-alloy ratio." In the process of molding this amalgam into test specimens, some of the mercury, along with a small amount of alloy, is squeezed out by the pressure exerted through the piston of the mold. The percentage of mercury in the finished test specimen is here designated as the "mercury content."

<sup>6</sup> For convenience, nearly all pressures are here expressed in terms of kilograms-weight per circular centimeter. 1 kg. per cir. cm. =  $4\pi$  kg. per cm.<sup>2</sup> = 18.05 lb. per in.<sup>2</sup> Black and his followers express crushing strength in terms of pounds-weight under which a 0.085-in. cube failed. Such a result must be multiplied by 7.6 to reduce to kilograms-weight per circular centimeter.

The time of packing was found to exert but little influence upon the crushing strength,<sup>7</sup> especially when the packing pressure was low. Our earliest experiments indicated that it would be safe to standardize upon a packing time of 8 min. in order to eliminate possible irregularities arising from this source. Later tests under controlled temperature

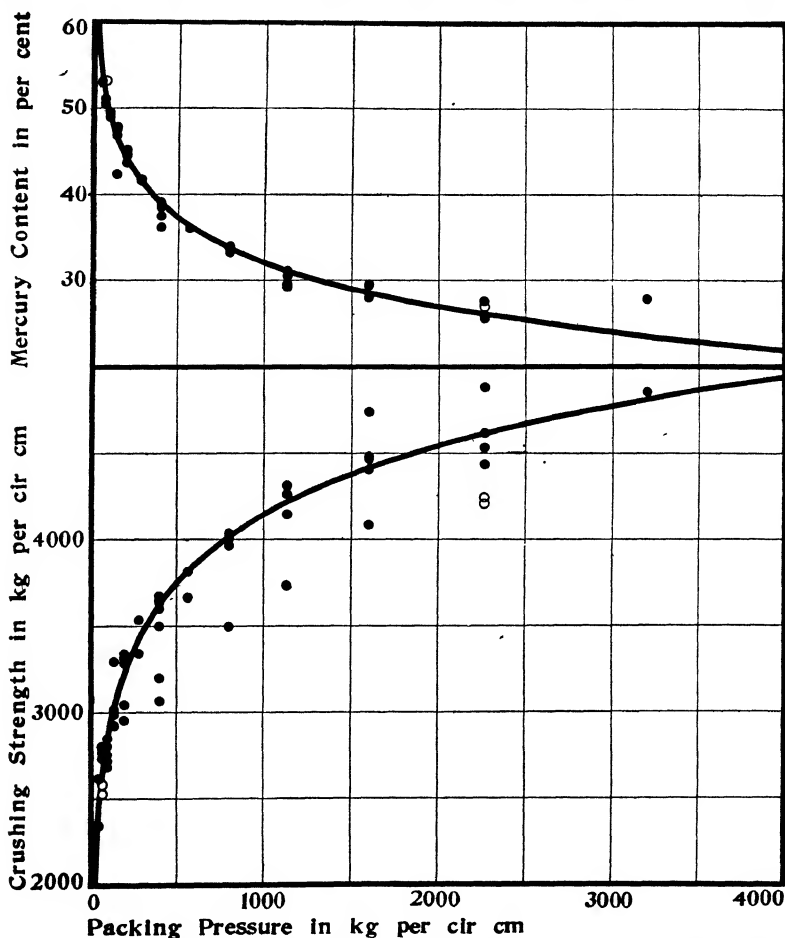


FIG. 6.—BOTH CRUSHING STRENGTH AND MERCURY-CONTENT OF AN AMALGAM VARY AS LOGARITHM OF PACKING PRESSURE. LARGE DEVIATIONS FROM LOWER CURVE ARISE FROM CRUSHING SPECIMENS AT VARIOUS TEMPERATURES.

showed the advisability of reducing the time to 4 min.; still later ones resulted in adopting 2 min. as the standard. The slight increase of strength observable when a high packing pressure was maintained for

<sup>7</sup> Throughout this paper the term "crushing strength" is used to designate the pressure (almost always expressed in kg. per cir. cm.) under which the test specimen failed when subjected to simple axial compression.

a considerable time seemed traceable to cold working. This view was strengthened by results obtained at higher temperatures. Dilatometric measurements of reaction expansion (which were found to be far more delicate indicators than results of crushing tests) showed but little change upon reducing the packing time to one-quarter of a minute (see p. 689).

The packing pressure was found to exert a marked influence (Fig. 6). Even the earliest tests at room temperature showed that, other things being equal, the crushing strength  $S$  increases directly as the logarithm of the packing pressure  $P$ ; so that, within the limits of experimental error, it can be expressed by a formula of the type  $S = A + B \log P$ , which is equivalent to  $P = e^{aS-b}$ , in which  $e$  stands for the logarithmic base, while  $A$ ,  $B$ ,  $a$ , and  $b$  are parameters depending upon such factors as the nature of the amalgam and the temperature of crushing. At the same time, the mercury content was found to decrease logarithmically with increasing packing pressure; so that it, too, could be represented by a similar formula.

The curves shown in Fig. 6 were calculated from such equations. It can be seen that nearly all the points representing the results of the individual tests lie close to the curves. A few, however, deviate by more than appears to be the uncertainty of our crushing tests. As was then assumed, and now confirmed by later experiments, these irregularities can be readily explained by the fact that the tests were made at room temperature on different days during the summer, which resulted not only in different cylinders being crushed at widely different temperatures, but also in different amounts of moisture being condensed from the air upon the filings of alloy as they were being triturated with mercury. In addition, some of the deviations may have been due to the use of hand power in applying the crushing load, and to those occasional irregularities that seem inseparable from strength tests, such as defective specimens and failure to adjust properly between the jaws of the testing machine.<sup>8</sup>

The alloys used in all the other tests described in the second part of this paper were likewise amalgamated by trituration in a mortar, unless the contrary is stated. In most cases, however, 6 gm. of alloy were used, and changes were made in such factors as mercury-alloy ratio, trituration time, and age of the amalgam when tested.

The logarithmic law connecting strength with packing pressure now has the support of more than 4000 crushing tests of amalgams prepared

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<sup>8</sup> Each cylinder used in the tests recorded in Fig. 6 was prepared from 5 gm. of a high-grade alloy in the form of filings, just as furnished to the dentist. This was incorporated with 1.40 times its mass of purified mercury by thoroughly triturating in a glass mortar for 1.5 min. The resulting amalgam was quickly rolled into a ball, dropped into the mold, and packed immediately. The cylinders were crushed 24 hr. after making.

under widely varying conditions. The only systematic deviations from it thus far observed were found with cylinders undergoing transition: either the mix had not completely hardened, or else it was softening from the transformation that occurs near 70° C. (see p. 671). Even in these cases the deviations were only slight.

#### APPLICATION OF LOGARITHMIC LAW

As soon as the law was discovered it was applied for checking and adjusting observations. Instead of following the usual engineering procedure of averaging the results from several tests of specimens all subjected to the same treatment, cylinders for testing a given point were always prepared in groups of three, or of five, packed under pressures increasing in geometrical progression. The pressures actually used were from the series 100, 141, 200, 283, 400, 566, 800, 1131, and 1600 kg. per cir. cm., in which  $\sqrt{2}$  is the common ratio. Then, if the crushing strengths found for all the cylinders of a given group were plotted as equidistant ordinates, the points obtained should lie close to a straight line. This procedure yields just as good an average value as if all the cylinders of the group were packed under the same pressure. In addition, it yields the particular curve connecting strength with packing pressure that holds for the conditions under which the test is made. Its justification lies in the fact that, with the exceptions noted in the preceding paragraph, the logarithmic law has always been found to represent the observations within the limits of experimental error.

#### INFLUENCE OF AMALGAMATION VARIABLES

The fact that both the strength and the mercury content change regularly with the packing pressure suggests at once a close connection between the former and the latter. Long ago, Black found that the more mercury he squeezed out during the packing of a given amalgam, the stronger was the filling he obtained; but his methods of experimenting were not sufficiently precise to bring out any quantitative relationships. That the suggested connection is not of the nature of cause and effect, but is rather a coincidence resulting from a common cause, becomes apparent as we notice how strength and mercury content are affected by varying such factors as the mercury-alloy ratio and the trituration time.

Figs. 7, 8, and 9 exhibit the results of varying the mercury-alloy ratio from 0.5 to 2.5, while the trituration time was maintained uniformly at 1.5 min. This varied the mix from a very stiff to a very pasty one. The cylinders packed under the lowest pressure (141 kg. per cir. cm.) show the phenomenon most clearly, both mercury content and crushing strength increasing rapidly in the same general way to maximum values,

which remain constant as the mercury-alloy ratio is still further increased. The illustrations show how the increases of packing pressure progressively wipe out the effects and cause the maxima to be reached sooner; also, that constant mercury content is reached before constant strength.

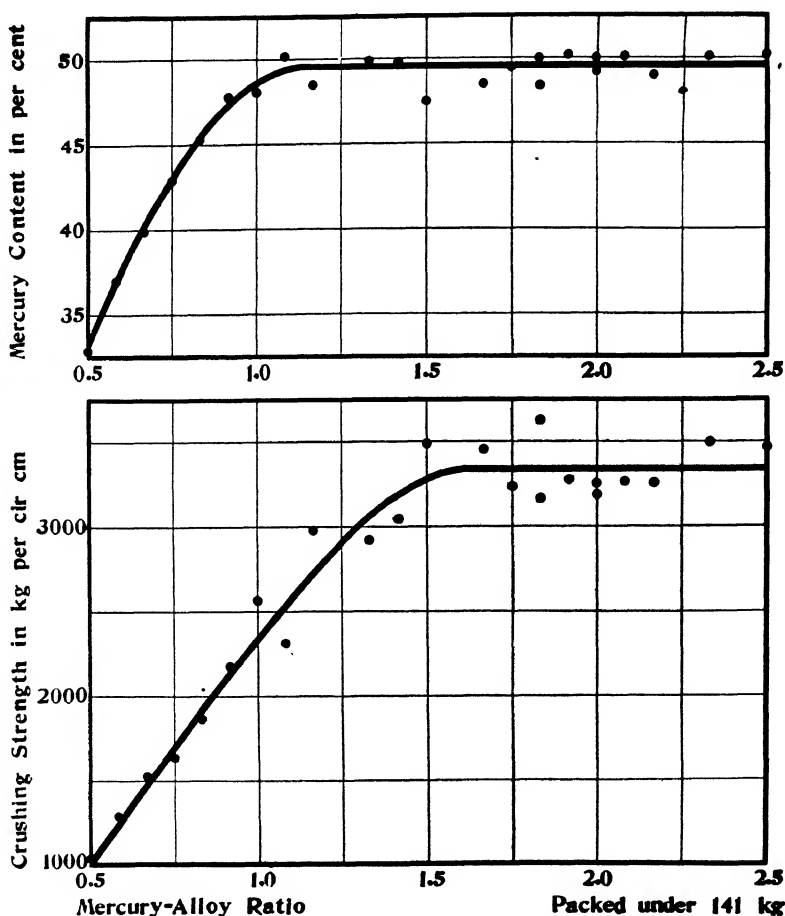


FIG. 7.—EFFECTS OF VARYING RELATIVE AMOUNTS OF MERCURY AND ALLOY THAT ARE MIXED TOGETHER IN MAKING AN AMALGAM.

Figs. 10 and 11 exhibit the results of varying the trituration time from 1 to 8 min., while a constant mercury ratio of 1.40 was used. The curves of Fig. 10 show that, in general, more mercury is retained in a test cylinder by prolonging the trituration; but it is interesting to note that when the packing pressure is low, less mercury seems to be left in the cylinder by increasing the trituration time from 1 to 2 min. Apparently this is because the shorter trituration leaves many of the alloy granules so

large that a low packing pressure is insufficient to squeeze out the free mercury from the spaces among the solid particles. The curves also show that for a given trituration time the mercury content changes almost inversely as the logarithm of the packing pressure.

Increasing the trituration time while the packing pressure is kept constant is accompanied by a progressive increase in strength (Fig. 11)

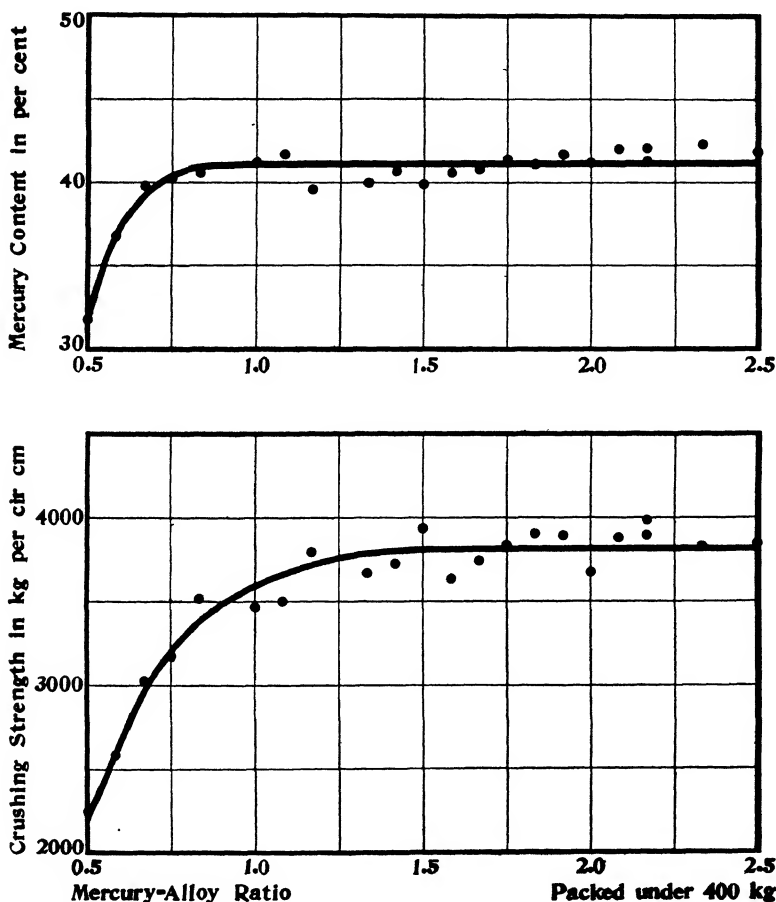


FIG. 8.—MODIFICATION OF FIG. 7 PRODUCED BY INCREASING PACKING PRESSURE FROM 141 TO 400 KG. PER CIR. CM.

until the latter reaches a maximum when the trituration is maintained for about 6 min. Prolonging the time beyond this brings about a very gradual falling off in strength on account of the incipient setting of the amalgam during the mixing. The results also indicate that the logarithmic law connecting crushing strength and packing pressure is applicable for any given trituration time within the range investigated.

The reason why excessive amalgamation produces so little effect upon the mercury content would appear to be simply that the metals entering into a dental alloy are only very slightly soluble in mercury.<sup>9</sup>

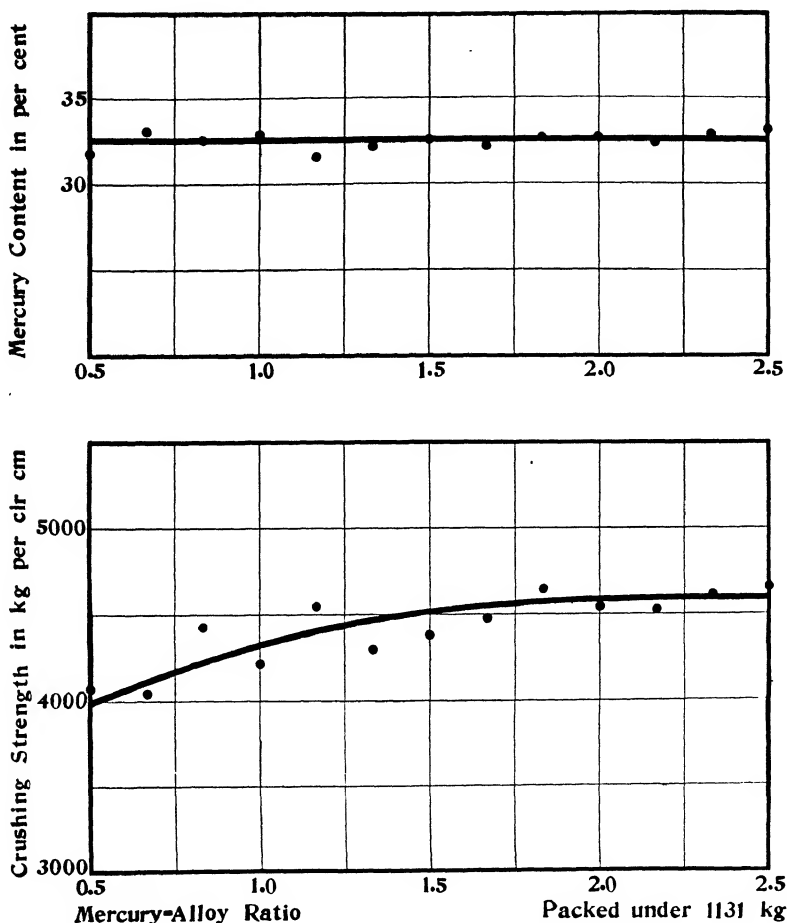


FIG. 9.—MODIFICATION OF FIGS. 7 AND 8 BY FURTHER INCREASE OF PACKING PRESSURE TO 1131 KG. PER CIR. CM.

<sup>9</sup> McBain and Joyner (*Journal, Chemical Society of London* (1911) **99**, 195–208; *Dental Cosmos* (1912) **54**, 646–648) analyzed mercury that had been saturated by two weeks' contact in a thermostat with filings of silver-tin alloys, ranging by 10-per cent. steps from one pure metal to the other. They found that at 25° C. the mercury dissolved but 0.044 per cent. silver and 0.75 per cent. tin, and that these saturation values were independent of the composition of the alloy. At 63° C., about 0.18 per cent. and 2.5 per cent., respectively, were dissolved. Even the most thorough amalgamation of dental office practice is hardly likely to bring about complete saturation.

# INFLUENCE OF TEMPERATURE

Fig. 12 represents the results of crushing 63 similar cylinders of a high-grade dental amalgam at temperatures fairly uniformly distributed

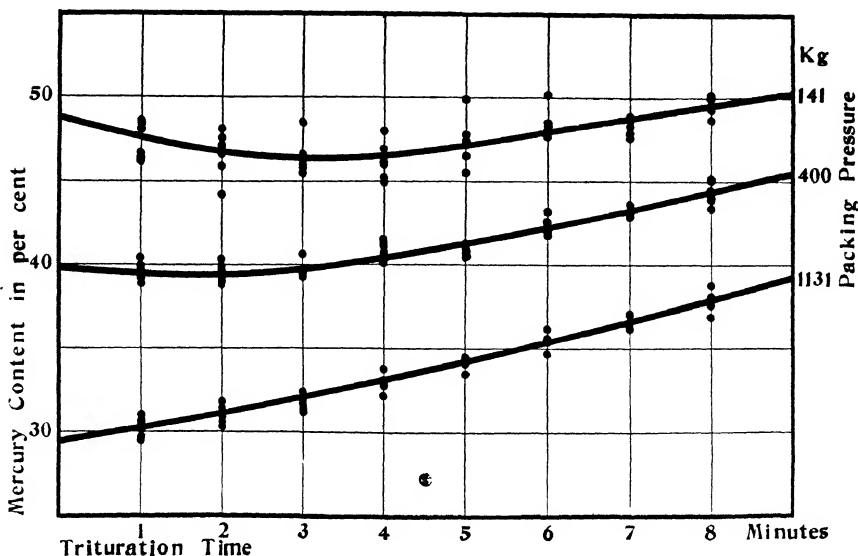


FIG. 10.—CHANGES IN MERCURY CONTENT PRODUCED BY VARYING TIME DEVOTED TO TRITURATING MIX OF ALLOY AND MERCURY.

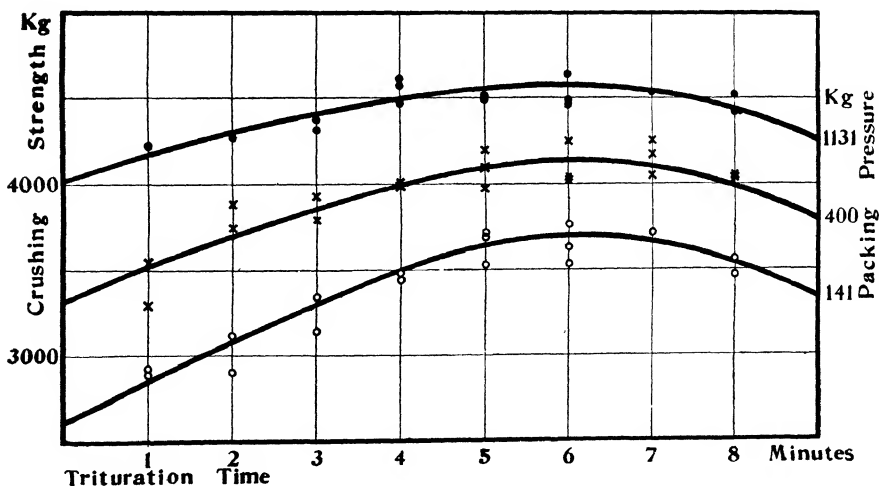


FIG. 11.—GAIN IN CRUSHING STRENGTH PRODUCED BY INCREASING TRITURATION TIME.

over the entire range from below 25° C. to over 95° C. Each cylinder was prepared by incorporating the same mass of alloy filings with 1.60 times



this mass of mercury. After triturating for 4 min., the resulting smooth, plastic amalgam was packed under a load of 400 kg. maintained for 8 min. This produced a cylinder 10.04 mm. in diameter by 11.5 mm. high, 40 per cent. of its mass being mercury. All the cylinders prepared in this way were immediately placed in an incubator kept at 37.5° C.

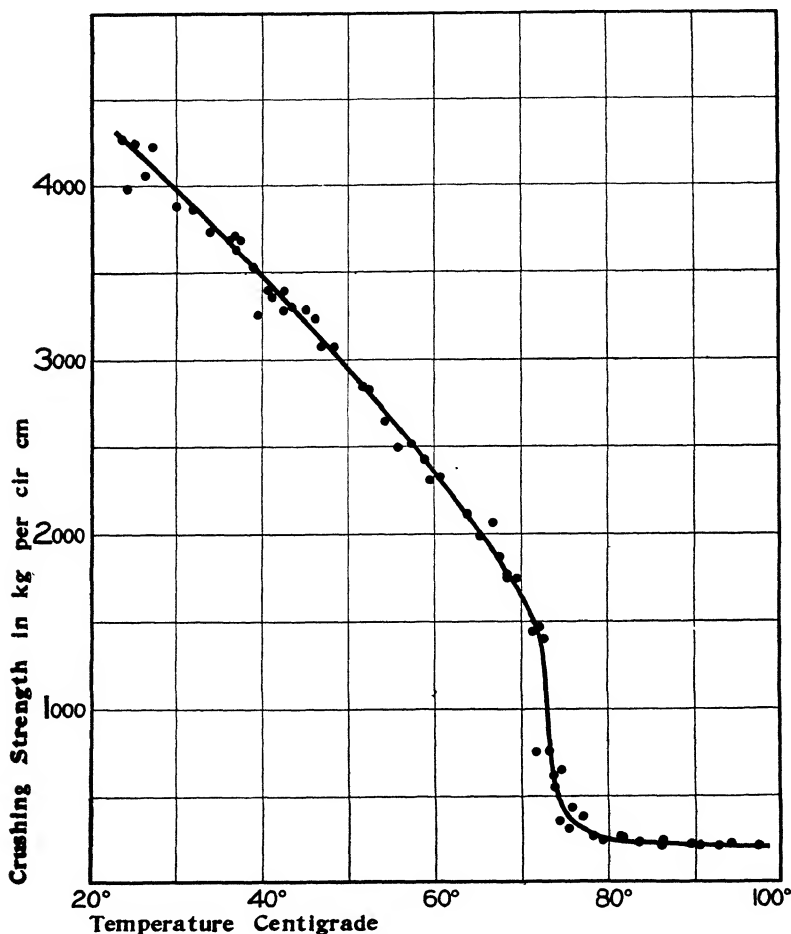


FIG. 12.—EFFECT OF TEMPERATURE ON CRUSHING STRENGTH. TRANSITION REGION SHOWN BY THE RAPID FALL IN STRENGTH BETWEEN 70° AND 80° IS CONFIRMED BY FIGS. 13 AND 14.

(body temperature), where they remained for several days before crushing, thus insuring practical completion of the hardening process. The alloy used contained approximately 68 per cent. silver, 26 per cent. tin, 5 per cent. copper, and 1 per cent. zinc.

All the 63 individual determinations made on three separate days

are included in the chart. The abscissa of a point represents the temperature of a cylinder at the time it was being crushed; the ordinate, the compressive load in kilograms-weight sustained by the cylinder at the instant of failure, and, therefore, the crushing strength in kilograms-weight per circular centimeter. The closeness with which the points follow a smooth curve shows the uniformity with which a dental amalgam can be prepared, and also the precision with which crushing tests can be made, provided proper precautions be taken.

The curve indicates that with rising temperature the crushing strength of the particular amalgam represented decreases somewhat faster than linearly from 5300 kg. per cm.<sup>2</sup> at 25° to 4050 at 45°, and 2550 at 65°. At 37.5° the strength was found to be 4550 kg. per cm.<sup>2</sup> or nearly 65,000 lb. per square inch.

#### TRANSITION REGION NEAR 70° C.

One of the most interesting features of this curve is the sudden plunge that it takes soon after passing 70° C. Black had observed that an amal-

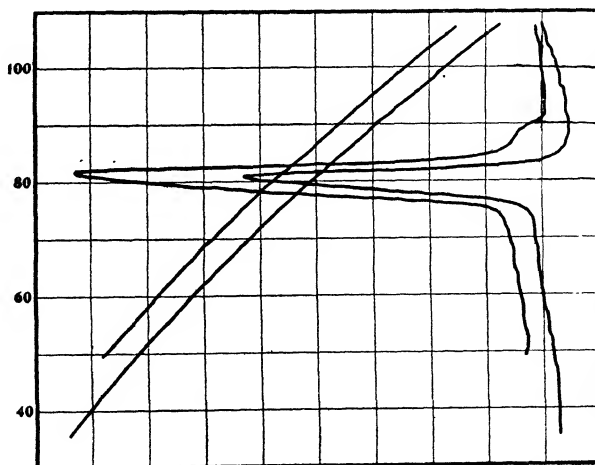


FIG. 13.—TRANSITION REGION REVEALED BY MODIFIED ROBERTS-AUSTEN METHOD. TEMPERATURES PLOTTED AS ORDINATES ARE THOSE OF NEUTRAL BODY. ABCISSAS ARE GALVANOMETER DEFLECTIONS. DIAGONAL CURVES SHOW RATE OF HEATING NEUTRAL BODY, EACH HORIZONTAL SPACE REPRESENTING 20 MIN.

gam, upon heating to the temperature of boiling water, suffered a noticeable permanent change in both appearance and volume, which he surmised was caused by something other than ordinary thermal-expansion.<sup>10</sup> McBain and Joyner, employing a liquid dilatometer in the usual way,

<sup>10</sup> *Dental Cosmos* (1895) 37, 661.

located a sharp transition at  $71.5^{\circ}\text{C}$ .<sup>11</sup> I have confirmed the existence of this transition by means of two other entirely independent methods of thermal analysis.

Fig. 13 reproduces typical curves obtained by a modification of the Roberts-Austen method of observing temperature differences between the sample and a neutral body while both were being heated simultaneously in the same enclosure. It is evident that the transition is accompanied by an appreciable absorption of heat.<sup>12</sup> The heating curves obtained with two cylinders packed for 8 min. under 141 and 1131 kg. per cir. cm., respectively, showed sharp peaks at essentially the same temperature, which was close to  $77.3^{\circ}\text{C}$ ., corresponding to about the

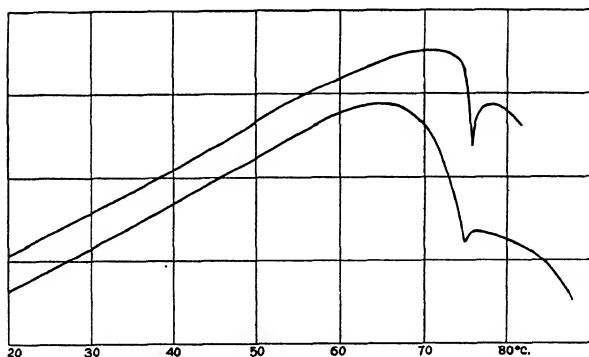


FIG. 14.—TRANSITION REGION REVEALED BY DILATOMETRIC METHOD. CURVES REPRESENT HEATINGS OF SAME SPECIMEN ON TWO SUCCESSIVE DAYS.

completion of the sudden drop in strength. The magnitude of the heat absorption was somewhat greater with the cylinder packed at the lower pressure, which also contained a greater proportion of mercury. The beginning and the end of the transformation are not sharply defined, but are in the neighborhood of  $70^{\circ}$  and  $90^{\circ}$ , respectively. On cooling from  $100^{\circ}$  to  $50^{\circ}$  no corresponding evolution of heat was observed, showing that the transition is not a reversible one. The absorption of heat, however, reappears at about the same temperature with each successive warming of the same specimen; but the magnitude of the effect progressively diminishes.

Fig. 14 reproduces a pair of curves obtained by a method that depends upon variations in linear thermal expansivity. The two curves show

<sup>11</sup> *Ibid.* (1912) 54, 649.

<sup>12</sup> McBain and Joynér (*Ibid.*, 54, 647) state that "Comprehensive series of cooling curves of the various amalgams were taken, but the chemical changes involved are too slow for this method to be advantageous. It was found that all amalgams of tin and silver, even those that have long hardened, undergo partial fusion at  $65^{\circ}$  to  $100^{\circ}$ , and gradually become more fluid as the temperature is raised."

the effect of heating the same specimen on successive days. The modifications produced by the first heating and the reappearance of the sharp minimum should be noted. The amalgams represented by Figs. 13 and 14 were prepared from different alloys.

#### INFLUENCE OF PACKING PRESSURE AT VARIOUS TEMPERATURES

Having learned how the strength of a high-grade dental amalgam could be altered by varying separately either the packing pressure or

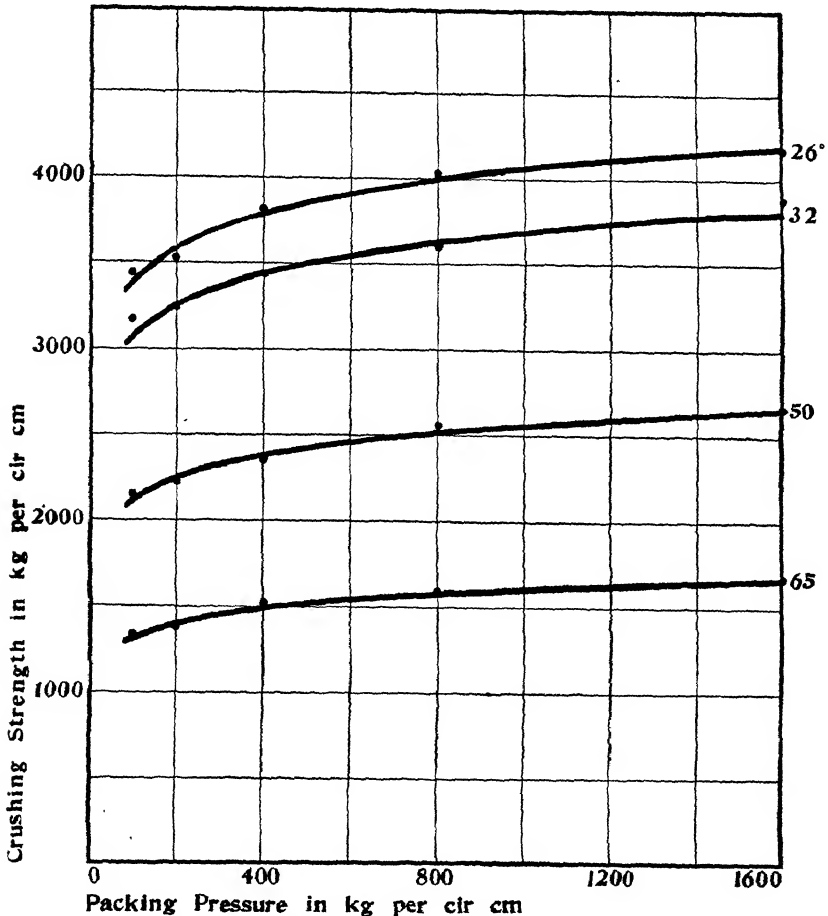


FIG. 15.—LOGARITHMIC LAW CONNECTING CRUSHING STRENGTH AND PACKING PRESSURE HOLDS AT ANY TEMPERATURE BETWEEN 26° AND 65° C.

the temperature of crushing, it became of interest to investigate the effect of simultaneously varying both of these parameters within wide limits. Accordingly, a group of test cylinders were packed for 4 min. under

loads of 100, 200, 400, 800, and 1600 kg. per cir. cm. After storing at 37.5° for 3 days, these were crushed at temperatures distributed over the interval from 25° to 100° C.

Some of the results are represented by Fig. 15. The curve for each temperature was calculated from an equation of the type  $S = A + B \log P$ , in which the letters have the same meaning as on p. 664. The

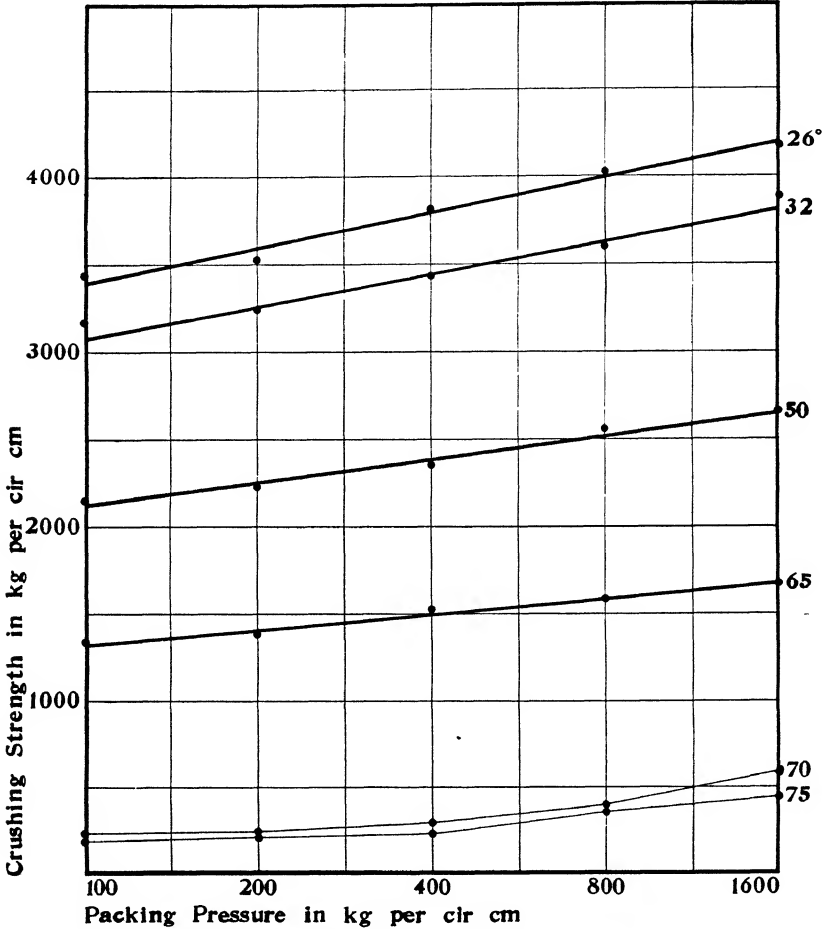


FIG. 16.—TRANSFORMATION OF LOGARITHMIC CURVES OF FIG. 15 BY PLOTTING ABSCISSAS PROPORTIONAL TO LOGARITHMS OF PACKING PRESSURES.

plotted points, each of which represents an individual crush (not an average of several tests), were located after applying small corrections to reduce the observations to what they would have been if the temperatures at the time of crushing had been exactly those stated along the right-hand margin.

Precisely the same data are plotted in Fig. 16. The only difference is that the curves of Fig. 15 have been transformed into straight lines by the simple mathematical expedient of making the abscissas proportional to the logarithms of the packing pressures. The results of tests at 70° and at 75°, which are here added, indicate slight deviations from the

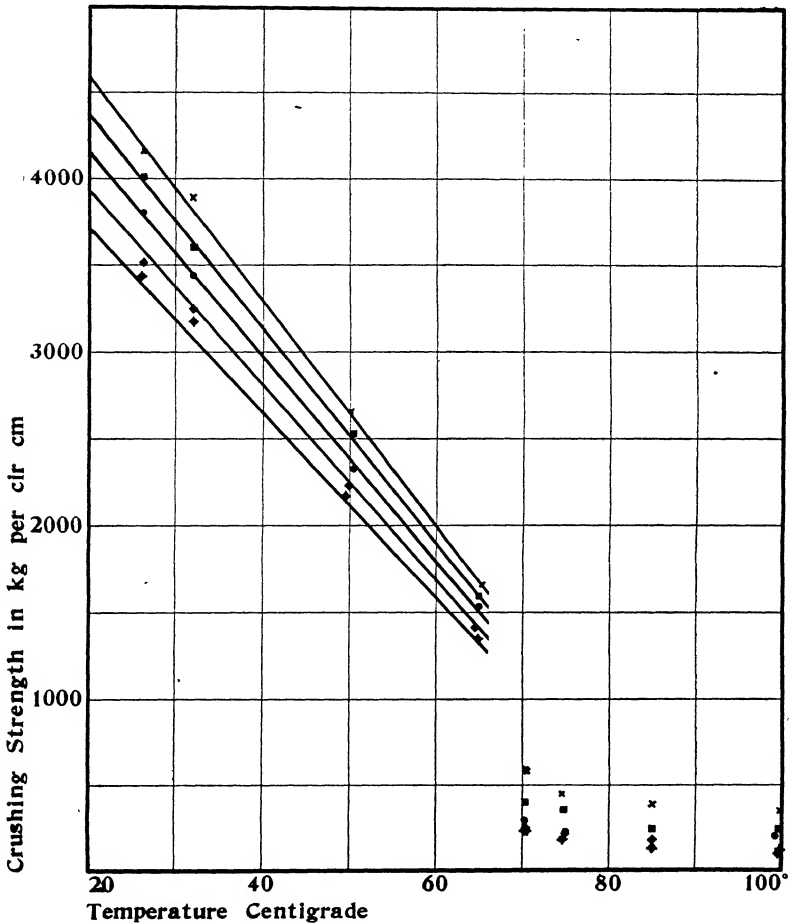


FIG. 17.—DATA REPRESENTED IN FIGS. 15 AND 16 PLOTTED TO SHOW THAT STRENGTH OF GIVEN AMALGAM SPECIMEN DECREASES LINEARLY WITH INCREASE OF TEMPERATURE. LOGARITHMIC LAW IS EXPRESSED BY EQUAL ANGULAR DIVERGENCE OF STRAIGHT LINES CORRESPONDING TO VARIOUS PACKING PRESSURES.

logarithmic law at these temperatures, but these deviations are hardly greater than the experimental errors.

Still another view of these same facts is afforded by Fig. 17. Here again the straight lines (each of which corresponds to a definite packing pressure, ranging from 100 kg. per cir. cm. at the bottom to 1600 at the

top) result from direct mathematical transformation of the lines appearing in the preceding two figures, the logarithmic relations being expressed by the equal angular spacing. In this plot no corrections are necessary for temperature deviations from the nominal values. The points represent the actual observations, each packing pressure being indicated by its own symbol. Additional observations near  $85^{\circ}$  and  $100^{\circ}$  are included.

It is possible that the lines representing the results between  $25^{\circ}$  and  $65^{\circ}$  in Fig. 17 should be slightly curved as in Fig. 12. However, the straight lines, as drawn, afford a simple means of expressing the observed facts within the limits of experimental error. The reason why the first part of the curve in Fig. 12 lies above the median line of Fig. 17 is to be sought in the fact that the cylinders used for the former were somewhat harder, because they were subjected to the packing pressure for 8 min. instead of 4, and they were stored more than twice as long before crushing.

Cooling the amalgam below  $20^{\circ}$  or  $25^{\circ}$  was found to result in a marked reduction of the rate at which strength changes with change of temperature. Perhaps this has some connection with the transition of tin from the white to the gray form.

#### INFLUENCE OF AGE UPON ALLOY AND AMALGAM

Two more questions of vital interest to the dentist will now be considered. (1) How does the strength vary with the time that elapses

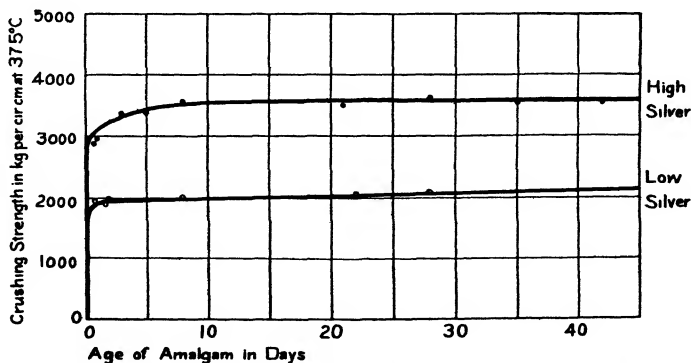


FIG. 18.—HARDENING OF AMALGAMS. AMALGAM FROM HIGH-SILVER ALLOY IS 75 PER CENT. STRONGER THAN THAT FROM LOW-SILVER ALLOY.

after the plastic mix is condensed into a rigid cylinder by the application of the packing-pressure? Does an amalgam, after hardening to its maximum strength, maintain this strength indefinitely? (2) Does the alloy from which the dentist prepares his amalgam deteriorate from long standing on the dealer's shelves?

In Fig. 18, the upper curve shows the behavior of an amalgam from

the same high-grade alloy that has been represented in the preceding charts; the lower curve shows the behavior of an amalgam from a typical low-grade alloy still much used by dentists all over the country. Both alloys were treated in exactly the same way; the cylinders had been packed

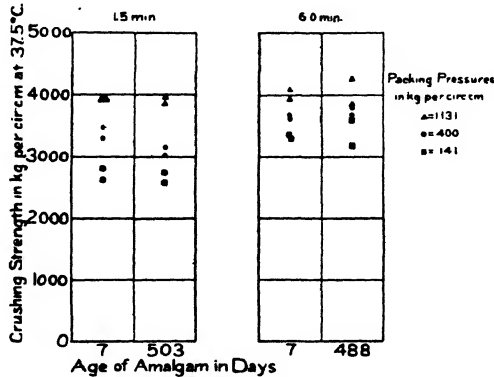


FIG. 19.—AMALGAMS FROM DENTAL ALLOY OF GOOD QUALITY SHOW NO RETROGRESSION IN STRENGTH DURING MORE THAN  $1\frac{1}{2}$  YEAR.

for 2 min. under 400 kg. per cir. cm. The results with other packing pressures yielded similar curves, which are here omitted to avoid confusing the chart. For the same reason, those cylinders between 0.1 hr. and 6 hr. old are also omitted. Note the extremely rapid rise in

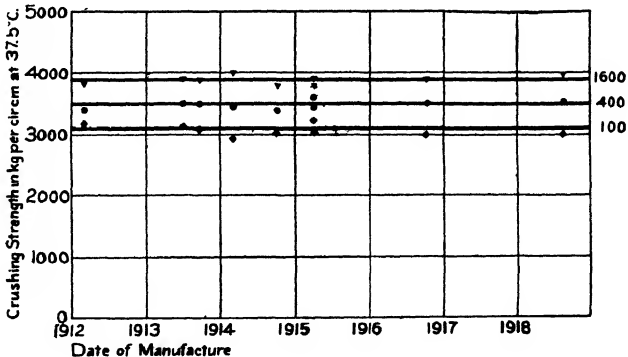


FIG. 20.—UNIFORMITY AND STABILITY OF DENTAL ALLOY OF GOOD QUALITY FIGURES TO RIGHT OF LINES INDICATE PRESSURES UNDER WHICH AMALGAMS WERE MOLDED INTO TEST CYLINDERS.

strength during the first 2 hr., followed by a slow increase that continues for more than a week. Note, also, the striking difference between the high-grade and the low-grade alloy.

The stability of the high-grade amalgam is well brought out by Fig. 19, which compares cylinders only 7 days old with others which had



been stored at body temperature for more than 16 months before crushing. Whether the mix was triturated for 1.5 or for 6 min., whether it was packed under 100, 400, or 1600 kg., there is not the slightest indication of retrogression in strength. The results demonstrate conclusively that it is perfectly possible to make a dental alloy which will yield a stable amalgam.

Fig. 20 shows the uniformity of product attainable in the regular commercial manufacture of a high-grade dental alloy. It shows, also, that the sample of alloy which had been standing in the package as long as 6 years yielded just as strong an amalgam as the sample which had been made only a few days before testing. These results prove beyond question that a properly made alloy does not deteriorate with lapse of time, provided it has been kept reasonably clean and has not been subjected to gross abuse such as storing in an unusually warm place.<sup>13</sup>

#### DIMENSIONAL CHANGES DURING THE HARDENING OF AN AMALGAM

I must now turn to the consideration of those highly interesting and important volume changes that take place during the hardening process, changes that for lack of a better term I shall call "reaction expansions."<sup>14</sup>

#### BLACK AMALGAM MICROMETER

The instrument, commonly known as the Black amalgam micrometer, shown in Fig. 21, is essentially a form of spherometer for measuring movements in the free surface of an amalgam filling packed into a cavity.<sup>15</sup> This cavity is bored into one face of a round steel block, called a Wedelstaedt test tube. In the foreground of the picture can be seen a number of these which contain fillings, the tube in the upper right-hand corner

---

<sup>13</sup> Ward conducted an extensive series of tests which led him to the conclusion that some, at least, of the high-silver dental alloys on the market show marked deterioration with time both before and after being made into amalgam fillings. *Dental Review* (1907) **21**, 416; *Dental Cosmos* (1908) **50**, 114-116; *Journal, Allied Dental Societies* (1916) **11**, 446.

<sup>14</sup> Changes in microstructure continue for a long time after the amalgam is packed into the tooth, and these changes bring about not only rapid increase of strength and hardness but also dimensional changes, which, however, have nothing to do with the ordinary thermal expansion that follows change of temperature. They are accompaniments of the internal readjustments that take place, even at constant temperature, and may properly be called "reaction expansions," using the term in the algebraic sense to include both expansion and contraction. During hardening an amalgam may either expand or contract. More often, it moves first one way, then the other. It is not intended that the use of the term will imply that the phenomenon is regarded as caused by a purely chemical reaction. The term is used for convenience to cover all dimensional changes that occur during hardening at constant temperature, whatever may be the nature of the internal processes that bring them about.

<sup>15</sup> *Dental Cosmos* (1895) **37**, 637 ff.

of the group being empty. The black dots in the centers of the fillings are thin steel disks embedded in the amalgam while still soft, in order to prevent the measuring contact of the micrometer from sinking in. One of the test tubes is shown as placed in the instrument.

From the standpoint of the professional metrologist, the design of the Black micrometer is interesting as an example of accuracy in some parts, combined with abundant opportunity for serious errors, both systematic and accidental—errors which neither Black nor his followers seem to have suspected. While the dial under the pointer of the instrument is graduated in divisions corresponding to 0.0001-in. change in elevation of the amalgam surface, and readings are commonly expressed to one-quarter of this unit, the results cannot be relied upon to any such extent.

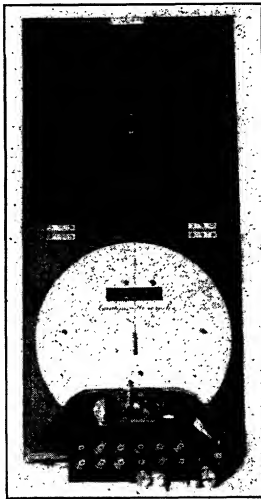


FIG. 21.—BLACK AMALGAM MICROMETER WITH GROUP OF WEDELSTAEDT TEST TUBES IN FOREGROUND.

I have nowhere been able to find the slightest indication that any of the users of amalgam micrometers ever thought to calibrate his instrument. The only check that seems to have been applied consisted in examining the filling margins with a binocular microscope, which, of course, would reveal nothing until the contraction or the expansion was great enough to tear the amalgam away from the cavity walls.

While the design of the Black amalgam micrometer is such as to render it unreliable for measurements requiring high accuracy, that of the Wedelstaedt test tubes is still more unsuitable. What is measured by the micrometer is neither linear nor volumetric expansion; it might appropriately be called heaped-measure expansion. Even when a contraction is indicated, the movement of the filling is not a simple linear dis-

placement until the amalgam has become completely free from the constraints imposed upon it by the walls of the cavity. The only reliable procedure for accurate measurements of expansions—whether thermal or reaction—is to employ a method which leaves the specimen perfectly free to change its dimensions in the direction of the displacements under examination.

The combination of Black micrometer and Wedelstaedt test tubes gives opportunity for still other uncertainties. A slight tipping or loosen-

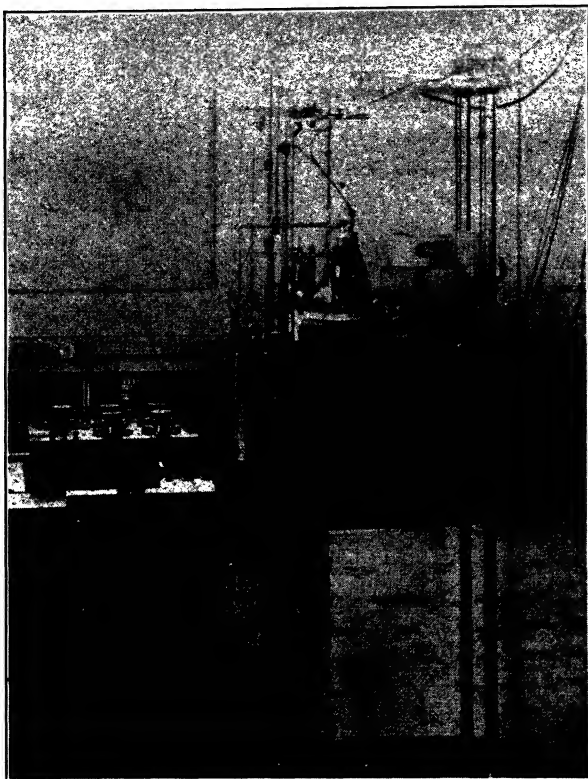


FIG. 22.—THERMOSTATED DILATOMETER DESIGNED FOR ACCURATELY MEASURING DIMENSIONAL CHANGES DURING HARDENING OF AMALGAM AT CONTROLLED TEMPERATURE.

ing of the little disk against which the measuring point presses has been known to give totally misleading readings. The pressure exerted by this contact point is far too great. The readings obtained depend too much upon the way the instrument is manipulated. Lack of temperature control, and the irregularities that are inseparable from specimens packed by hand pressure or by mallet blows in the ordinary dental way, still further confuse the results. It is no wonder, then, that we find

officially reported<sup>16</sup> to the National Dental Association results of Black micrometer tests on the same high-grade alloy that range from 1 to 20 points expansion.<sup>17</sup> Even one of Black's own pupils in alloy-making, after 20 years of almost daily experience with the instrument, found the results entirely too unreliable for making further progress in the manufacture of an alloy of the highest grade. And no other instrument of the accuracy<sup>18</sup> required for the refinements contemplated had yet been invented. It was for this reason that I was called upon to design suitable instruments and to reinvestigate the whole problem with all the facilities now available for metallographic research.

#### THERMOSTATED DILATOMETER DESIGNED BY AUTHOR

An exterior view of one of the instruments that I designed for studying reaction expansions appears in Fig. 22. It is a thermostated linear dila-



FIG. 23.—THREE VIEWS OF AMALGAM CYLINDER USED IN MEASURING REACTION EXPANSIONS TO AN ACCURACY OF 0.05 MICRON. NATURAL SIZE.

tometer capable of measuring displacements with an accuracy of 0.05 micron =  $1/500,000$  in., which is one-fiftieth the least count of Black's amalgam micrometer. Moreover, it is provided with arrangements for cooling and for heating, so that a specimen under observation can be maintained at any desired temperature from below  $-50^{\circ}$  C. to over  $+300^{\circ}$  C.

The amalgam specimens used with this dilatometer are molded into short, right, circular cylinders 1 cm. in diameter. Three views of such a

<sup>16</sup> *Dental Cosmos* (1913) **55**, 57 and 59.

<sup>17</sup> One "point" expansion represents a movement of 0.0001 in. = 2.5 microns in the measuring point that presses against the specimen. As a means for describing a characteristic of the amalgam it is, however, extremely indefinite; partly for reasons given in connection with Wedelstaedt tubes, partly because the dimensions of the cavities in these tubes do not conform to any fixed standard. Black (*Dental Cosmos* (1896) **38**, 967), in defining a "point" as 0.0001 in., says, "Its relative value in shrinkages or expansions is one in three thousand, lineal measurement," an idea which he repeats on p. 975, where he gives the dimensions of the cylindrical cavity as 0.38 in. in diameter by 0.30 in. in depth. Elsewhere (*Ibid.* (1895), **37**, 639) he gives 0.38 by 0.38. On the next page he gives  $\frac{3}{8}$  by  $\frac{1}{4}$ . McCauley (*Ibid.* (1913) **55**, 56) gives  $0.33 \times 0.38$ .

<sup>18</sup> By "accuracy" is meant all that this word implies; not "least" count, which can easily be made as small as one pleases by merely increasing the multiplying factor (and at the same time increasing the errors) of the instrument; nor even "precision" of repeating a given measurement. "Correctness," "reliability," are meant.

cylinder are shown in Fig. 23. The small axial holes molded into each base are for supporting the specimen between centers, so that it will be perfectly free to expand or contract radially. The radial groove near the circumference of one base is for engaging the pointed end of a screw which prevents the cylinder from turning on its axis. This insures that the contacts for transmitting the displacements to the multiplying mechanism will always bear against the ends of the same diameter. The cylinders are prepared just as those used for crushing tests (p. 661). This method very accurately makes all the cylinders of the same diameter, so that the insertion of a fresh cylinder never requires the readjustment of the instrument to bring the scale within the field of the telescope by which it is read. Moreover, the cylindrical surfaces are so smoothly formed that reliable bearings of the measuring contacts are assured.

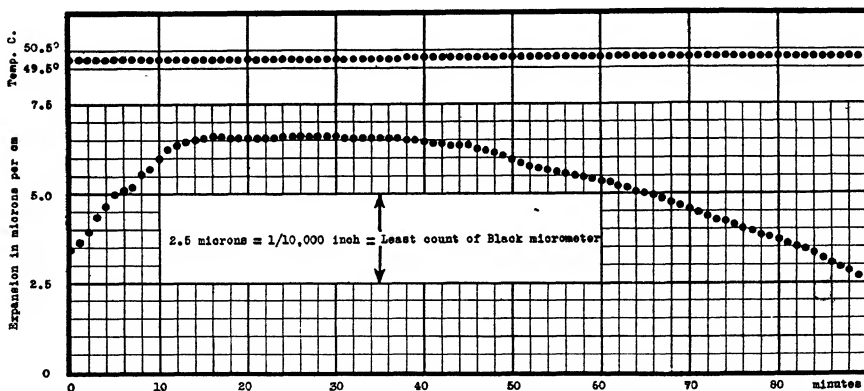


FIG. 24.—GRAPHIC REPRESENTATION OF REACTION EXPANSIONS. DETAIL OF CURVE SHOWN IN FIG. 25 BETWEEN BLACK DOTS. INDIVIDUAL OBSERVATIONS MADE WITH DILATOMETER EVERY MINUTE WHILE DETERMINING DIMENSIONAL CHANGES THAT OCCUR DURING HARDENING OF A DENTAL AMALGAM. INSTRUMENT RELIABLE TO 0.05 MICRON =  $1/500,000$  IN., WHICH IS  $1/50$  LEAST COUNT OF BLACK MICROMETER. DURING MEASUREMENT WITH DILATOMETER, AMALGAM IS MAINTAINED AT DESIRED TEMPERATURE BY THERMOSTATED STIRRED OIL-BATH.

The multiplying mechanism is a combination of mechanical and optical levers so designed that the pivotal and the frictional errors almost always present in such arrangements are eliminated. A simple optical device eliminates errors that would otherwise proceed from those minute changes in the relative positions of telescope, mirror, and scale that are constantly taking place in all such apparatus. My instrument, which is in daily use, is so little sensitive to external disturbances that nothing short of a direct blow will vitiate its readings 0.05 micron. Still, the moving parts are so delicately adjusted that the measuring contacts that bear against the amalgam can be separated by such a small force as that produced by pressing sidewise with the tip of a camel's hair brush.

The amalgam dilatometer is used for following the course of reaction expansions from minute to minute throughout the day. Measurements can be started within 1 min. after a cylinder has been molded; but the first two or three observations are likely to be affected by small errors

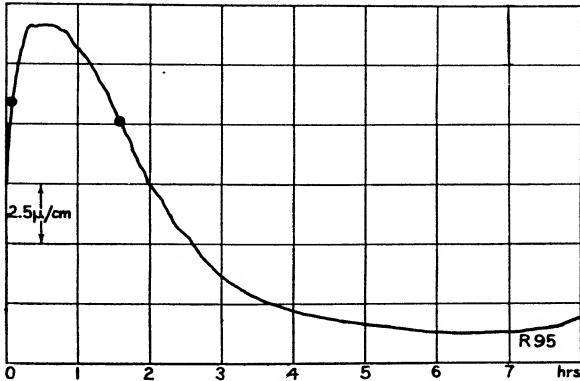


FIG. 25.—CURVE REPRESENTING DIMENSIONAL CHANGES OCCURRING IN DENTAL AMALGAM DURING FIRST 8 HR. OF HARDENING.

caused from disturbing the temperature when the oil-bath is lowered long enough to permit the insertion of the specimen.

#### GRAPHICAL REPRESENTATION OF REACTION EXPANSIONS

A series of individual observations made 1 min. apart are plotted in Fig. 24. This represents the results obtained during only  $1\frac{1}{2}$  hr. of an

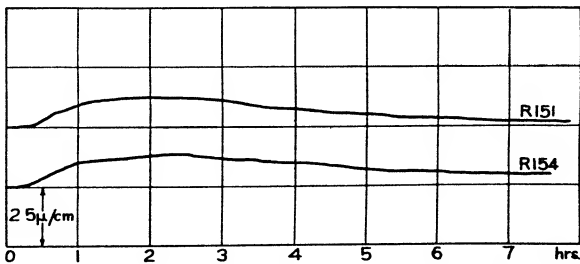


FIG. 26.—SHOWING RELIABILITY WITH WHICH TWO CYLINDERS OF AMALGAM SAME ALLOY CAN BE PREPARED AND MEASURED.

8-hr. run. In order to include the entire series of a day's observations in a single chart of convenient size, the original plot is made on the scale of  $\frac{1}{40}$  in. to the minute, which brings the points so close together that they must be represented in the published charts as continuous curves (drawn by tracing the original plots). Such a curve is shown in Fig. 25.

The part included between the two prominent dots represents exactly the same observations as the lower chain of dots shown in Fig. 24. The upper chain of dots represents the temperature observations. Since the temperature of the dilatometer bath seldom varied more than  $0.1^{\circ}$ , its record is not shown in subsequent charts. In all of these, the base of a small rectangle represents 1 hr., the altitude an expansion of 2.5 microns = 0.0001 in. (approximately) in the 1-cm. cylinder of amalgam.

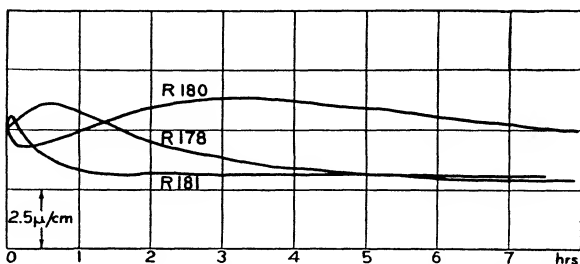


FIG. 27.—TYPICAL REACTION EXPANSION CURVE (R180) AND MODIFICATIONS CAUSED BY INCREASE OF PACKING PRESSURE.

Fig. 26, which represents the behavior of a different amalgam, is presented merely to show how accurately results can be reproduced with a given alloy. Cylinders *R 151* and *R 154* were made on separate days. The two curves obtained are almost exactly congruent. As will be seen later, this affords a most delicate test for the uniformity of the technique employed in preparing the amalgam cylinder from the filings of alloy, as well as for the reliability of the dilatometer in recording such minute displacements.

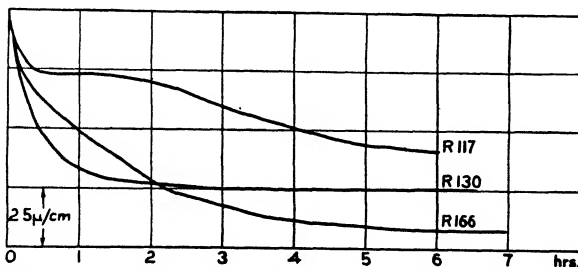


FIG. 28.—VARIATIONS OF CURVES SHOWN IN FIG. 27 PRODUCED BY INCREASING TRITURATION TIME.

### TYPICAL REACTION EXPANSION CURVE

From a group of several hundred curves representing reaction expansion in a dental amalgam under various conditions, a few are selected to illustrate certain general principles. Contrary to the generally accepted teachings of Black, the dimensional changes of an amalgam

from the same alloy can be very profoundly altered by altering the manipulation.<sup>19</sup>

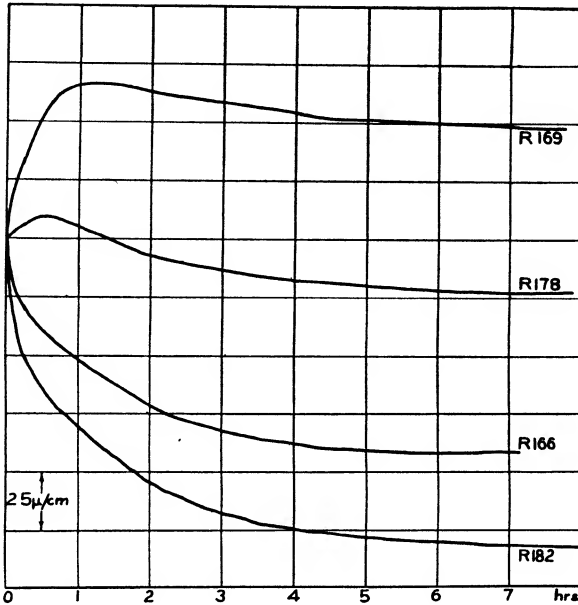


FIG. 29.—EFFECTS PRODUCED BY VARIATION OF TRITURATION TIME.

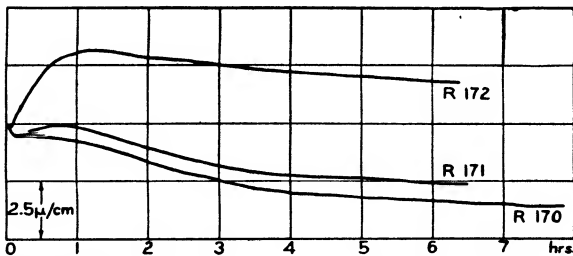


FIG. 30.—TRANSFORMATION OF CURVES OF FIG. 29 BY REDUCING TEMPERATURE FROM 37.5° TO 25° C.

In the curve representing the behavior of specimen *R* 180, Fig. 27, there is a slight contraction followed by a slow expansion, which in turn

<sup>19</sup> Black states that "Shrinkage and expansion of the amalgam used is not under the control of manipulation by the operator. This can be controlled only in the selection of the alloy to be used. Shrinkage or expansion is controlled by the proportions of the metals of which the alloy is formed, and their relations to each other as alloyed or mixed. In the particular compound selected for use, shrinkage or expansion does not depend upon the proportion of mercury; further than that, wide shrinkages or wide expansions can in some degree be modified as to extent, not controlled. As these are only modifications and do not control, they are unimportant in the manipulative sense; the question of shrinkage and expansion resolves itself exclusively into that of the correct balancing of the metals in the alloy." "Operative Dentistry," 2, 319. Chicago, 1908. Medico-Dental Pub. Co.



is followed by still slower contraction. This is the typical reaction expansion curve for a dental amalgam. This assertion does not mean that all the curves actually obtained look more or less like this one, but rather that every curve so far obtained may be regarded as one of a continuous family, the individual members of which result from progressively transforming a curve of this shape. By appropriate modifications of the alloy and of the manipulation accompanying amalgamation we can produce all the gradations in form that separate one curve from another which at first sight seems to bear no relation to it. Some concrete examples will make this clear.

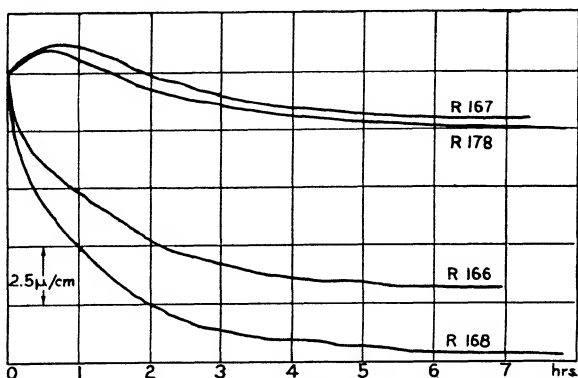


FIG. 31.—EFFECTS PRODUCED BY CHANGING MERCURY-ALLOY RATIO AND TRITURATION TIME.

#### INFLUENCE OF PACKING PRESSURE

The three specimens represented in Fig. 27 were prepared by triturating together the same proportions of the same alloy and mercury for the same time. All were packed for 2 min. in a mold at 37.5° C. and were kept at this temperature during the observation of their dimensional changes. The differences in shape are produced solely by changing

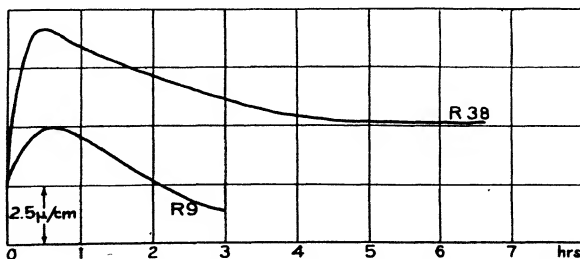


FIG. 32.—EFFECT OF LARGE CHANGE IN MERCURY-ALLOY RATIO.

the packing pressure from 100 kg. per cir. cm. with R 180 to 400 kg. per cir. cm. with R 178 and 1600 kg. per cir. cm. with R 181. The increases of pressure progressively shorten the time consumed in reaching the

stages corresponding to the minima and the maximum. The change from 100 to 400 kg. per cir. cm. accelerates the reaction to such an extent that the initial contraction observable with R 180 is over before observations can be commenced; it has apparently taken place in the mold. The change from 400 to 1600 kg. per cir. cm. produces a further acceleration of the reaction; so that the maximum, which was reached by R 180

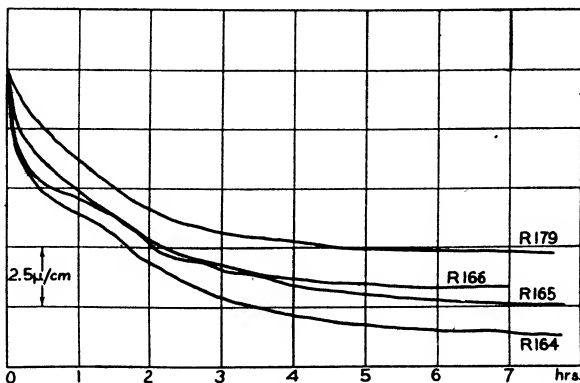


FIG. 33.—SMALL EFFECTS PRODUCED BY VARYING PACKING TIME.

only after the lapse of 3 hr., is reached by R 181 in about 4 minutes. Such an increase in the reaction rate is just what one might reasonably expect from the more intimate union between mercury and alloy that accompanies increase of packing pressure.

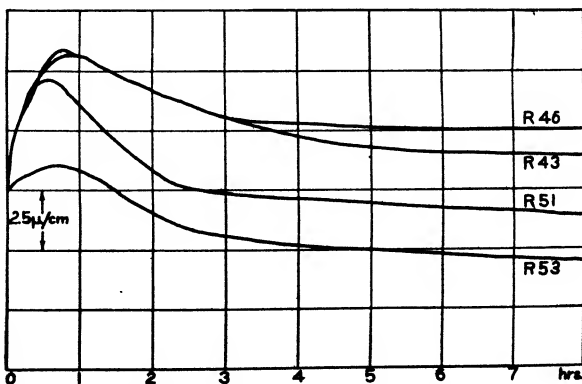


FIG. 34.—EFFECTS PRODUCED BY VARYING ANNEALING TIME OF ALLOY.

#### INFLUENCE OF TRITURATION TIME

Again, accelerating the reaction by increasing the trituration time from 1.5 to 4.0 min., while keeping all other conditions the same, changes the appearance of the curves from that shown in Fig. 27 to that shown in Fig. 28. R 180 has been transformed into R 117, R 178 into R 166,

and R 181 into R 130. In these the faint remains of the maxima so prominent in Fig. 27 may be seen. The progressive alteration in shape as the trituration time is progressively increased is, perhaps, still more clearly shown by Fig. 29, which reproduces R 178 and R 166, and adds two other curves for the same packing pressure of 400 kg., the first of which (R 169) shows the result of shortening the trituration time to 0.5 min., the second (R 182) the result of lengthening it to 6 minutes.

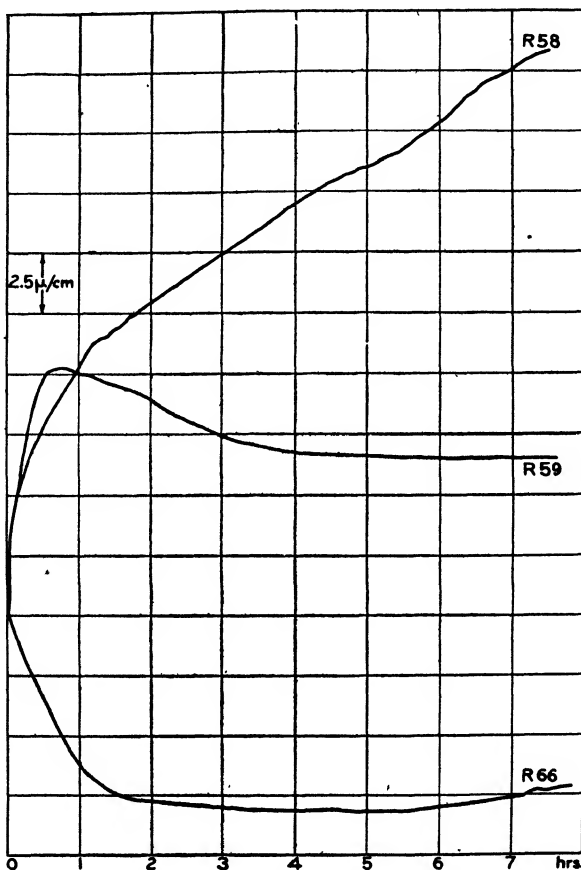


FIG. 35.—EFFECTS OF CHANGING SIZE OF ALLOY PARTICLES.

#### INFLUENCE OF TEMPERATURE

Fig. 30 shows how reducing the temperature from 37.5° to 25.0° C. transforms curves R 169, R 178, and R 166 into R 172, R 171, and R 170, respectively. This is merely an example of the familiar fact that reduction of temperature usually retards the rate of a chemical reaction. By cooling to 5 or 10° C. I have produced such a retardation that it

takes many hours to establish the existence of a slow contraction following the flat maximum.

### INFLUENCE OF MERCURY-ALLOY RATIO

Fig. 31 shows the effect of changing the mercury-alloy ratio. The amalgams for R 167 and R 178 were both triturated for 1.5 min.; but the mercury-alloy ratio was 1.40 in the former and 1.60 in the latter. The amalgams for R 166 and R 168 were both triturated for 4 min.; but the mercury-alloy ratios were 1.40 and 1.60, respectively. All four specimens were in other respects treated alike, being packed for 2 min. under

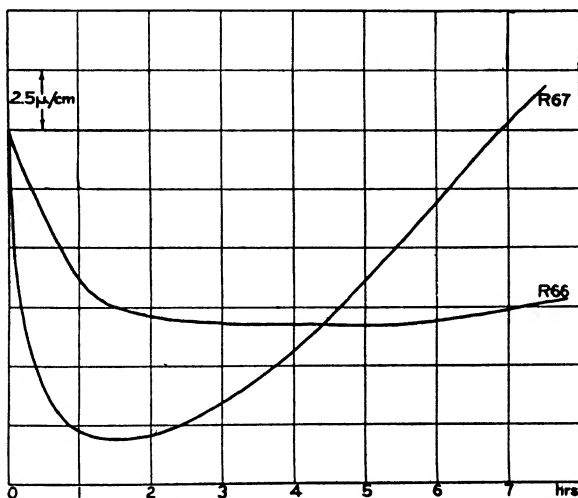


FIG. 36.—EXTENSION OF TYPICAL REACTION EXPANSION CURVE R 180 OF FIG. 27.

400 kg. and observed at 37.5°. When the mass of mercury is 1.40 times the mass of alloy with which it is mixed, it is considerably more difficult to obtain a smooth plastic amalgam than when the ratio of these masses is increased to 1.60. Increase of trituration time still further increases the plasticity. Fig. 31 shows how more thorough amalgamation actually changes the reaction rate in the direction in which we might naturally expect some change. Fig. 32 represents changes brought about by increasing the mercury-alloy ratio from the very low value of 0.60, used for R 38, to the very high value of 2.00, used for R 9. Both specimens were triturated for 1.5 min. and packed for 8 min. under 400 kg. The reaction expansions were measured at 50° C.

### INFLUENCE OF PACKING TIME

The small effect of packing time is shown by Fig. 33. All specimens were triturated for 4 min. with a mercury-alloy ratio of 1.60, resulting in

rather thorough amalgamation. All were packed under 400 kg. The time during which this packing load was applied varied from  $\frac{1}{4}$  min. to 8 min., as follows:  $\frac{1}{4}$  min. R 165, 1 min. R 164; 2 min. R 166; 8 min. R 179. The dimensional changes were measured at  $37.5^\circ$ . Some of the difference in the general positions of the curves is due to the fact that as the packing time is progressively increased, the time available for

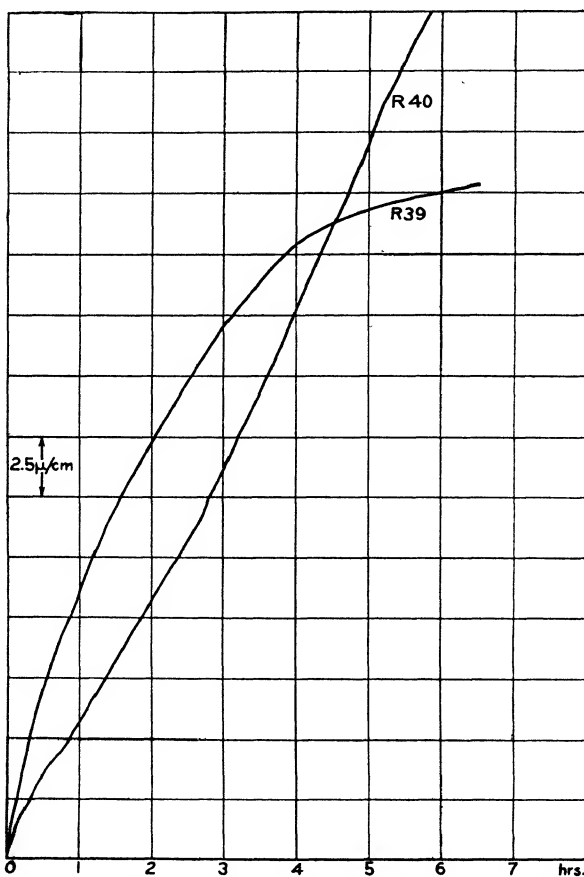


FIG. 37.—EFFECT OF SOFTENING A HARDENED AMALGAM BY HEAT (R 39) AND OF REAMALGAMATING (R 40).

observation in the dilatometer is decreased. This results in some obliteration of the initial steep descents in the curves.

All of the transformations that have been illustrated by Figs. 27 to 33, inclusive, were brought about by controllable changes in the manipulation of amalgams made from the very same batch of alloy, that is to say, by changes which must necessarily creep into any procedure for testing dental alloys for dimensional changes during hardening. It

is clear, therefore, that unless all influencing conditions are properly controlled, the results of such a test lead to misleading conclusions. There are still other transformations that may be produced by purely physical treatment of the alloy before it is mixed with mercury to form an amalgam.

#### INFLUENCE OF ANNEALING ALLOY

Annealing is an important instance of physical treatment. The temperature and the time required to anneal an alloy in order to produce the best product are of great importance in its manufacture. But these cannot be known until we know precisely how annealing modifies the properties of amalgams made from various alloys. Fig. 34 shows some

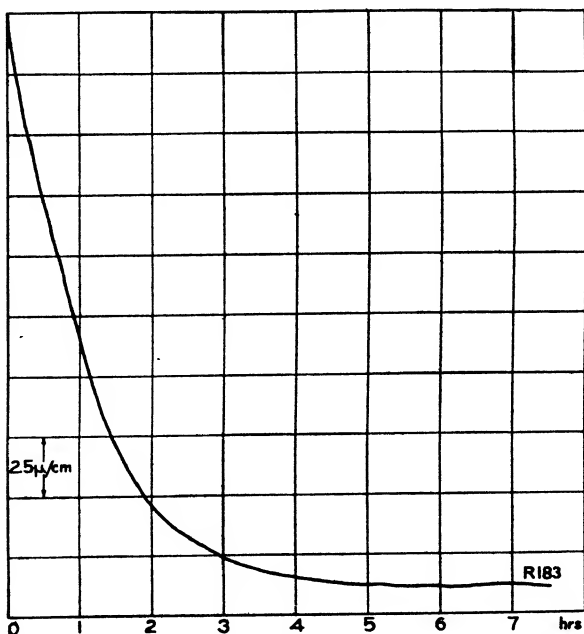


FIG. 38.—AMALGAM FROM A LOW-SILVER ALLOY. EXCESSIVE CONTRACTION.

effects of annealing the alloy represented by Figs. 27 to 33. R 53 was annealed for 5 days, R 51 for 10 days, R 46 for 15 days, and R 43 of 25 days.

#### INFLUENCE OF SIZE OF ALLOY PARTICLES

The size and shape of the alloy particles influence the reaction expansion. The three curves shown in Fig. 35 were obtained from the same batch of alloy filings manipulated in exactly the same way, as far as

amalgamation, packing, and temperature are concerned. But the particles used for *R* 58 were too coarse to pass through a sieve of 48 meshes to the inch, those used for *R* 59 passed through one of 200 meshes, and those used for *R* 66 were extremely fine. As the alloy is made progressively finer, the expansion curve undergoes the same sort of transformation that accompanies increase of trituration time (Fig. 29) and increase

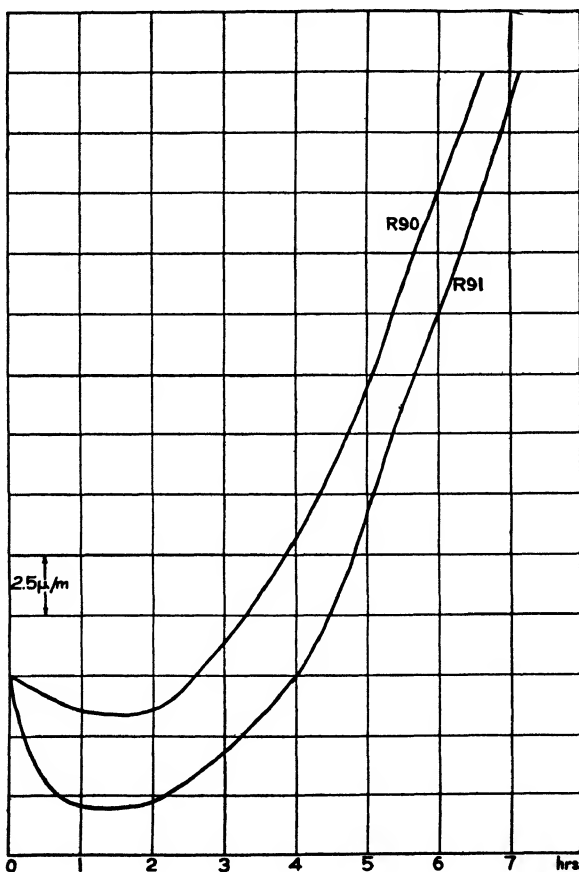


FIG. 39.—AMALGAMS FROM ANOTHER LOW-SILVER ALLOY. CONTRACTION FOLLOWED BY EXCESSIVE EXPANSION.

of mercury-alloy ratio (Fig. 31); that is to say, reducing the size of the alloy particles facilitates the amalgamation, a result that we might have expected in kind if not in degree. But curve *R* 66, after reaching its minimum, takes a slight upward turn, suggesting that there may be still another maximum beyond the second minimum of our type expansion curve *R* 180 of Fig. 27.

## EXTENSION OF TYPICAL EXPANSION CURVE

By progressively increasing the thoroughness of amalgamation, more and more of the early portions of the type curve are wiped out. At the same time the rate of reaction is increased so that the characteristic features make their appearance sooner and stand out more prominently. Therefore, if the slight rise at the end of *R* 66 really indicates

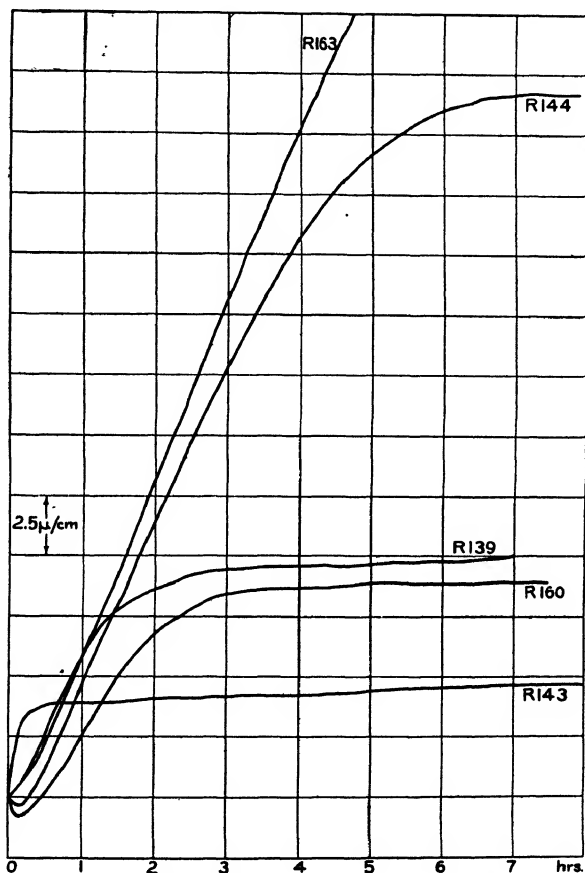


FIG. 40.—AMALGAMS FROM HIGH-SILVER ALLOYS.

the approach of another maximum, and not a mere vagary of the apparatus, more thorough amalgamation ought to emphasize this feature of the curve. Since the curves of Fig. 35 were obtained with a mercury-alloy ratio of only 1.40 and a trituration time of only 1.5 min., it was easy to test the matter. Fig. 36 shows how *R* 59 and *R* 66 were thus transformed into *R* 62 and *R* 67 upon increasing the mercury-alloy



ratio to 2.33 and the mixing to 6 min. trituration with mortar and pestle followed by 10 min. kneading in the hand.

# INFLUENCE OF THERMAL SOFTENING AND OF REAMALGAMATING A HARDENED AMALGAM

As a final example of transformations brought about by physical treatment of this same alloy, Fig. 37 is given. It shows results obtained by heating a hardened amalgam until it softens (*R* 39), and by mixing more mercury with filings from a hardened amalgam (*R* 40).

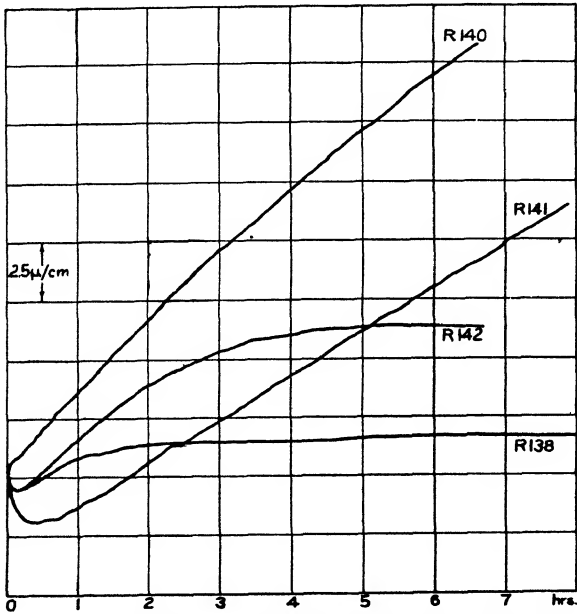


FIG. 41.—AMALGAMS FROM HIGH-SILVER ALLOY.

## CURVES OBTAINED WITH A VARIETY OF DENTAL ALLOYS

I shall conclude my exhibit of data by presenting without much comment a few curves obtained with amalgams from several different alloys. Figs. 38 to 44, inclusive, are typical. Figs. 25 and 26, which were introduced to illustrate precision of apparatus and technique, might be considered with this collection.

## COPPER AMALGAM

It may be of interest to point out that Fig. 44 reproduces two curves obtained with pure copper amalgam. Both of the specimens represented

were packed for 2 min. under 400 kg. per cir. cm. The dimensional changes were measured at 37.5° C. R 185 was triturated for 1.5 min., R 184 for 4 min.<sup>20</sup>

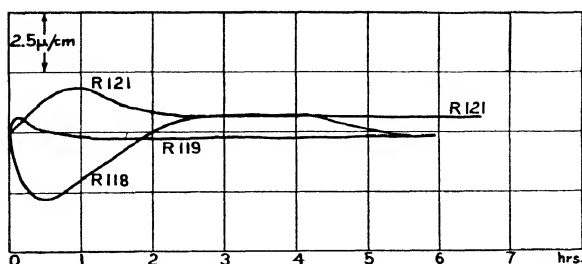


FIG. 42.—AMALGAMS FROM HIGH-SILVER ALLOY SHOWING CHARACTERISTIC CURVE AND MODIFICATIONS PRODUCED BY PROGRESSIVELY INCREASING PACKING PRESSURE (R 118, R 121, R 119). COMPARE WITH FIG. 27.

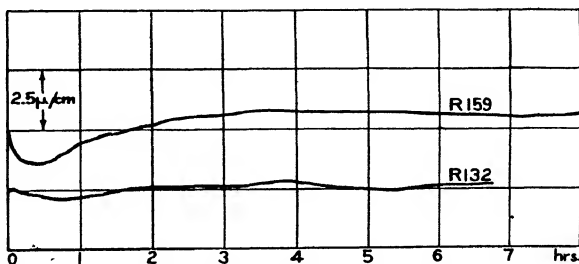


FIG. 43.—AMALGAMS FROM TWO HIGH-SILVER ALLOYS.

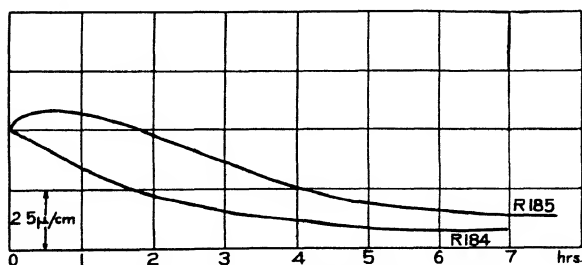


FIG. 44.—COPPER AMALGAM.

## PRACTICAL APPLICATIONS OF THE RESULTS

The determinations of both crushing strength and dimensional changes during hardening show how profoundly the behavior of an amalgam may

<sup>11</sup> Ward (*Journal, Allied Dental Societies* (1916) **11**, 457) says, "With the use of copper amalgam there seems to be a different phase of the problem confronting us at once. In the first place it does not change in volume to a degree that has been detected by any methods of measuring volume change at present known. It is supposed to be free from either contraction or expansion."

be influenced by such things as mercury-alloy ratio, trituration time, packing pressure, temperature during hardening, temperature during testing, and age of amalgam, as well as by the characteristics of the particular alloy under test. Therefore, comparisons of different dental alloys are worth little unless it is shown that proper attention has been given to the control of all such factors. It is just because such control is lacking that we must discard as worthless a great many of the tests that have been made with the Black dynamometer and with the Black and other micrometers. The traveling microscope, even though it be possible to repeat readings to within 1 micron or less, may conceal large instrumental errors, while the Wedelstaedt test tube is fundamentally wrong.

Contrary to general opinion, the soft, fine-grained amalgam obtained by long continued trituration with plenty of mercury hardens into a stronger filling than does a less plastic mix. By making the alloy so that the last movement shall be an expansion of desirable amount when a practicable packing pressure is employed, we secure a filling that approaches the ideal in regard to rapid hardening, final strength, plasticity, and volume changes. A fine-grained, plastic mass that expands slightly during hardening makes for tight fillings. Any procedure that brings about more intimate contact between the alloy particles and the mercury, and thereby accelerates the reaction, results in shortening the time required for the filling to reach its final, stable condition.

The production of a dental alloy to fulfill these requirements entails not merely the selection of a suitable formula, but also careful attention to various physical details of manufacture. A high-silver amalgam within a few hours becomes as strong as a low-silver amalgam ever becomes and finally attains a strength about 75 per cent. in excess of that attained by the low-silver. A given weight of a high-silver alloy makes a considerably larger filling than the same weight of a low-silver alloy, because it unites with more mercury, which is comparatively cheap. Consequently, it often proves more economical to use the high-silver alloy, which is superior from every point of view.

Two objections are sometimes raised against the use of a high-silver alloy. The first, that such alloys amalgamate with some difficulty, has been overcome by means of a recently invented process that removes from the annealed comminuted alloy surface impurities which hinder union with the mercury. The second, that a high-silver alloy requires more rapid manipulation, can be overcome by proper manipulation. After tritulating thoroughly with sufficient mercury to make a smooth, plastic mix, the dentist has merely to avoid expressing the excess mercury until just before placing the amalgam in the cavity. Moreover, an amalgam that has already begun to harden slightly may be softened

again without loss of strength by adding a little more mercury and working the mass until again plastic.

The form assumed by the type curve of reaction expansion, and the transformations it may be made to undergo by suitable control of the fundamental parameters, indicate the possibility of analysis into a group of superimposed curves of the exponential type, suggesting strongly a complicated reaction or transition that is separable into several elementary components which progress simultaneously and successively at different rates.

## Two Instances of Mobility of Gold in Solid State

BY EDWARD KELLER,\* PH. D., NEW YORK, N. Y.

(New York Meeting, February, 1919)

### GOLD MOVEMENT ON SURFACE OF AURIFEROUS COPPER WHEN LATTER IS SUBJECTED TO OXIDATION

SOME years ago the writer's attention was called to the fact that rolling-mill scales from auriferous copper do not have the gold content proportional to the gold contained in the copper from which they are derived.<sup>1</sup> So, later, he made a few simple experiments, which are described herewith.

In order to test the gold movement on the changing surface of a copper plate under the influence of oxidation, plates 6 in. by 8 in. by 1 in. (15.2 cm. by 20.3 cm. by 2.5 cm.) of converter copper, refined, of 99.29 per cent. purity, and leaded copper with a content of 95.65 per cent. copper, were obtained. Ten cuts were made through the thickness of the plates along the longitudinal center and the silver and gold content ascertained in each of the 10 samples in both plates, as shown in the accompanying illustration. The assay results, recorded in Tables 1 and 2, show that the gold distribution is relatively uniform in both plates, while silver has greater variations, especially in the leaded plate. The plates subjected to oxidation were placed in a red-hot muffle for about 2 hr., two small scorifiers being used as supports. Arranged alongside each other, they nearly filled the width of the muffle, and so were practically under equal conditions of heat and draft. The accurate observance of the time was not possible, however, because of other work, but the time of the exposure of each plate in a pair was identical. At the end of each period, each plate was plunged into water and completely freed of scale, which was carefully collected, dried, weighed, and analyzed. Samples of the plates had also been taken of the molten metal and duly analyzed.

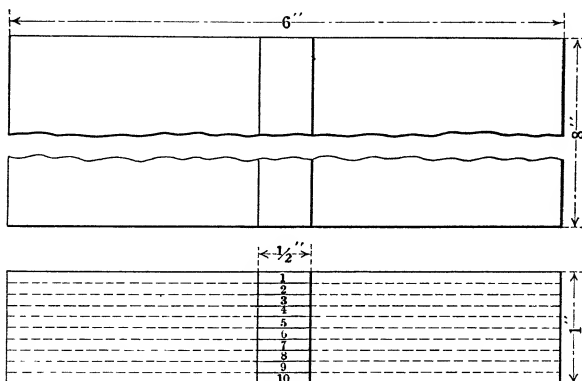
Table 3 contains the analytical data for the two plates and their several sheets, or layers, of oxide scales. These figures show that in the oxidations of the converter copper the silver is fairly uniform, while the gold is highly irregular and especially low in the first layer. In the oxidations of the leaded copper plate, the silver and gold contents are variable; however, this irregularity is due to two different causes. The

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\* Metallurgical chemist.

<sup>1</sup> H. A. Prosser, private communication.

fourteen oxidations with the converter copper plate and the five oxidations with the leaded copper plate left a residual plate approximately  $\frac{1}{4}$  in. (6.3 mm.) thick in each case.



METHOD BY WHICH SAMPLES WERE TAKEN.

TABLE 1.—*Converter Copper Plate*

Sample Number	Silver in Sample, Oz. per Ton	Gold in Sample, Oz. per Ton	Sample Number	Silver in Sample, Oz. per Ton	Gold in Sample, Oz. per Ton
1	77.1	0.34	7	75.6	0.32
2	76.4	0.34	8	75.8	0.34
3	76.1	0.34	9	77.4	0.34
4	76.5	0.32	10	77.3	0.34
5	76.3	0.34	Average	76.48	0.336
6	76.3	0.34			

TABLE 2.—*Leaded Copper Plate*

Sample Number	Silver in Sample, Oz. per Ton	Gold in Sample, Oz. per Ton	Sample Number	Silver in Sample, Oz. per Ton	Gold in Sample, Oz. per Ton
1	81.2	0.42	7	80.7	0.44
2	80.0	0.42	8	81.1	0.44
3	79.9	0.40	9	82.3	0.44
4	80.4	0.40	10	86.1	0.44
5	80.5	0.44	Average	81.27	0.428
6	80.5	0.44			

The figures given in Table 3 do not form a proper basis for comparison nor do they give the proper relation between the precious metals in the oxides and in the copper plates. The simplest way to show these rela-

TABLE 3.—Analyses of Copper Plates and Layers of Oxide Scales

Oxidation Sample Number	Converter Copper Plate				Leaded Copper Plate			
	Oxide, Grams	Copper, Per Cent.	Silver, Oz. per Ton	Gold, Oz. per Ton	Oxide, Grams	Copper, Per Cent.	Silver, Oz. per Ton	Gold, Oz. per Ton
1	410	86.93	68.20	0.040	1200	73.67	89.30	0.180
2	400	87.18	66.74	0.115	1110	78.79	66.84	0.245
3	550	87.71	67.75	0.125	1400	79.38	56.00	0.295
4	340	87.52	66.74	0.305	1040	79.32	60.28	0.215
5	390	87.83	65.94	0.110	800	78.91	54.52	0.230
6	580	88.01	64.65	0.230				
7	350	87.79	65.75	0.230				
8	435	87.96	64.76	0.215				
9	455	88.09	65.76	0.340				
10	420	87.77	66.01	0.415				
11	455	87.95	67.32	0.260				
12	395	87.60	65.31	0.300				
13	390	88.03	65.14	0.265				
14	375	88.00	64.90	0.285				
Plate...	....	99.29	76.30	0.315	....	96.65	81.00	0.410

TABLE 4.—Data in Table 3 Converted to Basis of Cu = 100

Oxidation Sample Number	Converter Copper Plate				Leaded Copper Plate			
	Copper in Oxide, Grams	Copper, Per Cent.	Silver, Oz. per Ton	Gold, Oz. per Ton	Copper in Oxide, Grams	Copper, Per Cent.	Silver, Oz. per Ton	Gold, Oz. per Ton
1	365.4	100	78.45	0.046	884.0	100	121.22	0.244
2	348.7	100	76.55	0.132	874.6	100	84.83	0.311
3	482.4	100	77.26	0.143	1111.3	100	70.55	0.372
4	297.6	100	76.26	0.348	824.9	100	76.00	0.271
5	342.5	100	75.08	0.125	631.3	100	69.09	0.291
6	510.5	100	73.46	0.261				
7	307.3	100	74.89	0.262				
8	382.6	100	73.63	0.244				
9	400.8	100	74.65	0.386				
10	368.6	100	75.21	0.473				
11	400.2	100	76.54	0.296				
12	346.0	100	74.55	0.342				
13	343.3	100	74.00	0.301				
14	330.0	100	73.75	0.324				
Average	373.8	...	75.30	0.2605	865.1	...	84.62	0.3024
Total...	5225.9	...	.....	.....	4326.1			
Plate...	.....	100	76.85	0.317	.....	100	84.68	0.428

tions is to transform these analytical figures to the basis of Cu = 100; the result is given in Table 4. The silver and gold is now directly comparable between each oxidation and the original copper plate, also

the silver and gold content in each oxide layer now pertaining to the copper that was oxidized. In the converter copper plate, the silver is fairly uniform in the several oxide layers and of about the same content as the average of the original plate; in other words, under the influence of oxidation of the copper, the silver is practically immobile. The gold is not only irregular but ranges from a minimum of 0.046 oz., in the first oxide layer, to a maximum of 0.473 oz., in the tenth layer. The correct average of the gold in all the oxidized copper of the converter copper plate is 0.2605 oz. per ton, or 82.2 per cent. of the 0.317 oz. per ton, which is the average gold for the whole plate. It follows, therefore, that at no stage of the oxidation was all the gold taken up by the oxide scales while some must have retreated and concentrated on, or in, the residual plate. For the leaded plate, Table 4 shows great variations in silver for the several oxidations. That these differences are not due to the mobility of the silver has already been indicated. Lead is not soluble in solid copper and in a copper-lead plate, like the one under consideration, most of the lead is retained mechanically on account of the rapid chilling after casting in a cold metallic mold. The mechanically contained lead appears to be under strain, for when the plate is reheated to redness the lead begins to ooze out at the top as well as the bottom, showing that other forces besides gravity are operative. Silver is more soluble in lead than in copper and moves from the inner plate outward with the molten lead as carrier. This fact was proved in a special test with a similar plate. The lead drops on the surfaces of the plate were collected before the copper had formed any appreciable oxide layer and their silver content was found to be 635.3 oz. per ton. The outward movement of the lead in the plate is confirmed, too, by the lower copper content of the first oxide layer, compared with the others, and by the higher copper content of the residual plate, compared with the original plate. The silver carried by the lead contains no appreciable amount of gold, so while the lead and the silver move outwards in the molten state, the gold concentrates toward the solid copper of the residual plate, even to a greater degree than in the case of the converter copper plate. The average gold content for all the copper oxidized in this plate is 0.3024 oz. per ton, or 70.65 per cent. of the average gold contained in the whole plate, which is 0.428 oz. per ton.

What has been proved of the gold movement toward the residual plates by the figures just given should also be proved by the analyses of the residual plates themselves. However, these residual plates, which were approximately  $\frac{1}{4}$  in. (6.3 mm.) thick, had samples planed from the top and bottom surfaces and these, as also one from the finally remaining plate, were analyzed, but the weights of the respective parts were not taken, so that a proper average of the assays cannot be given. Nevertheless, the results are given in Table 5.



TABLE 5.—Analyses of Residual Plates

Location of Sample	Converter Copper Plate			Leaded Copper Plate		
	Copper, Per Cent.	Silver, Oz. per Ton	Gold, Oz. per Ton	Copper, Per Cent.	Silver, Oz. per Ton	Gold, Oz. per Ton
Top surface.....	99.33	77.77	0 53	97.77	72.80	0 48
Bottom surface	99.38	75 35	0 35	97.70	72.30	0 58
Inner plate...	99 55	78 20	0.41	98.12	69.61	0.34

*Cast Copper, Quenched, Richer in Gold than Molten Charge*

Most of the converter copper now shipped from the smelteries in the form of anodes, bars, etc. is subjected to the so-called pickling process; that is, the red-hot castings are plunged into water, in which they shed most of the oxide scales that have formed on their surface. That the first layer of oxide, or scales, formed contains only about one-tenth of the gold in the original copper has been conclusively proved in the tests with the converter-copper plate. No data available show the commercial magnitude of this problem but the first layer of scales in the oxidation of the experimental converter copper plate may, perhaps, be an index of what takes place on a larger scale in metallurgical practice. The original plate was not weighed, but from its dimensions, 6 in. by 8 in. by 1 in., and the assay results the following data and conclusions are obtainable:

Weight of original plate, pounds.....	. 14 4
Weight of copper in first oxide scales (365.4 gm.), pounds..	0 8
Weight of remaining plate, pounds.....	13.6
Gold in original plate, ounces per ton..	0.315
Gold in copper of first oxide scales, ounces per ton	0.046
Weight of gold in original plate, ounces...	. 0.002268
Weight of gold in first oxide scales, ounces...	. 0.000018
Weight of gold in remaining plate, ounces	0.002250
Gold in remaining plate, ounces per ton. . . . .	. 0.331
Gold in original plate, ounces per ton . . . . .	. 0.315
Enrichment of remaining plate, ounces per ton... . .	0.016
	= 5 08 per cent.

This last figure, of course, will decrease with the increase of the thickness and weight of the plates or with the decrease in the thickness of the layer of oxidized copper. In the latter case, however, the scales would become poorer in gold, which would relatively increase the enrichment of the remaining plate. The thickness of the layer of copper oxidized in this experimental case approximates  $\frac{0.75}{28} = 0.027$  in.; where

TABLE 6.—*Monthly Averages of Western Shotted and Eastern Drilled Samples of Refined Converter Copper*

Month No.	Copper		Difference		Silver per Ton		Difference		Gold per Ton		Difference	
	Western Shotted Sample, Per Cent.	Eastern Drilled Sample, Per Cent.	Over, Per Cent.	Under, Per Cent.	Western Shotted Sample, Ounces	Eastern Drilled Sample, Ounces	Over, Ounces	Under, Ounces	Western Shotted Sample, Ounces	Eastern Drilled Sample, Ounces	Over, Ounces	Under, Ounces
1	99.3499	99.2922	.. . .	0.0577	88.6736	88.9444	0.2708	. . . .	0.5147	0.5226	0.0079	
2	99.3116	99.3077	.....	0.0039	83.4784	83.5366	0.0582	.....	0.4791	0.4903	0.0112	
3	99.2796	99.2864	0.0068	.	86.2927	86.2737	.....	0.0190	0.4958	0.5052	0.0094	
4	99.2730	99.2987	0.0257	0.0257	86.7758	86.8622	0.0864	. . . .	0.4738	0.4814	0.0076	
5	99.2525	99.2589	0.0064	. . . .	91.4387	91.4119	.....	0.0268	0.6244	0.6400	0.0156	
6	99.2621	99.2612	..	0.0009	91.3735	91.4701	0.0966	. . . .	0.5599	0.5665	0.0066	

0.75 in. is the thickness of all the copper oxidized and 28 the number of layers of scales produced by the 14 oxidations. It is not likely that this figure is reached in actual metallurgical practice.

Figures are available for copper sampled from the molten furnace charges and by drilling the pickled anodes. The samples by these methods are acknowledged to be correct and the assays were made by identical standard methods. Table 6 gives such a comparison on monthly runs for 6 mo.<sup>2</sup> The gold assays at the eastern end, made on drill samples from anodes, are uniformly higher than those of the west, where the assays were made on shotted samples from the furnace charges. These differences, though, are so small that they may be accidental or due to personal factors and render the point raised essentially academic; yet the factor undoubtedly exists and possesses the same algebraic sign at all times.

*Speed of Oxidation of Copper Containing Impurities*

The figures given plainly demonstrate that the copper of the leaded plate was much more quickly oxidized than the copper of the purer converter-copper plate and that the copper in the former was transformed approximately into cupric oxide while in the latter it remained essentially in the form of cuprous oxide. Table 7 gives the ratios of copper oxidized and of the oxygen taken up by the copper in the several plates. An additional test was made with a pair of plates, one of which was identical with the converter copper in the other tests, while the second contained approximately 1 per cent. of arsenic. The oxidation was

TABLE 7.—*Speed of Oxidation of Copper in Plates*

Number of Oxidation	Converter Copper Plate, Copper Oxidized, Grams	Leaded Copper Plate, Copper Oxidized, Grams	Copper Ratio	
			Converter Copper Plate	Leaded Copper Plate
1	365.4	884.0	1	2.42
2	348.7	874.6	1	2.51
3	482.4	1111.3	1	2.30
4	297.6	824.9	1	2.77
5	342.5	631.3	1	1.84
Total.....	1836.6	4326.1		
Average.....	367.5	865.2	1	2.35
Oxide, grams.....	2090.0	5383.5*	Oxygen Ratio	
Copper, grams....	1836.6	4326.1	1	4.17
Difference.....	253.4	1057.4		

\* Original oxide less 3 per cent. allowance for lead.

<sup>2</sup> E. Keller: Principles and Practice of Sampling Metallic Metallurgical Materials, U. S. Bureau of Mines, *Bulletin* No. 122 (1916) 16, 74

performed at a much lower temperature than in the former tests, therefore the smaller quantity of copper oxidized in about equal time. Of the converter copper, 73.79 gm. of copper were oxidized and 185.44 gm. of the arsenated plate, giving a ratio of 1 : 2.51.

Formerly copper sulfate (bluestone) was produced by oxidizing copper shot or granules in reverberatory furnaces and dissolving the finally obtained cupric oxide in sulfuric acid, etc. Comparatively pure and impure coppers were used, the latter having the advantage of much more ready oxidation. However, the disadvantage of the slow oxidation of the purer copper was overcome by converting it into hollow shot with very thin shell, thus enormously increasing its oxidizable surface. Pure copper, when molten, absorbs large volumes of sulfur dioxide, which is emitted upon cooling; therefore, when a jet of cold water, or air, strikes a stream of molten copper saturated with the sulfur dioxide the resulting shot are hollow. This absorbent power of pure copper for sulfur dioxide diminishes as other impurities increase and hollow shot cannot be produced from very impure copper.

#### *Explosive Condition*

In one of the series of tests, the two plates were left in the muffle for about double the usual time. When the converter copper plate was plunged into water, it shed its scales a little more noisily than usual but when the leaded plate was immersed, there was a loud report, the hand of the manipulator received a shock, and his eyes were firmly closed for some moments. The eyes, however, were absolutely uninjured and the very finely divided copper oxide that had struck the face with considerable force caused little irritation. The water bucket, though, was broken into numerous pieces.

There was an essential difference in the character of the scales of the two plates. Those from the converter-copper plate were relatively hard and remained in compact pieces of some size, while the scales from the leaded plate generally disintegrated into powder when striking the water. This suggests that the explosion was due to the enormous surface of the increased quantity of the red-hot oxide powder, spontaneously evolving a great volume of steam. This incident is recorded merely to call attention to what might happen if tons of similar material were plunged into a bosh.

#### GOLD MOVEMENT ON SURFACE OF AURIFEROUS SILVER WHEN LATTER IS DISSOLVED IN NITRIC ACID

It is generally known that in silver-gold alloys the silver is soluble in nitric acid when the alloy contains about 30 per cent. or less of gold and that in the higher grade of alloys comparatively strong nitric acid may be used without disintegrating the gold. As the gold content decreases and nitric acid above a certain strength is used, disintegration will take place. However, for the whole series of alloys down to 1 per cent. or even less of

gold, a proper strength of nitric acid and heat will dissolve the silver and leave the gold in such a coherent state that it represents the exact shape of the original bead, although very much reduced in size when little gold is present. Therefore it may be said that, with decreasing gold content in the silver-gold bead the more marked will be the gold movement in the direction of the retreating surface of the silver under the solvent action of the nitric acid.

#### *Bearing on Accuracy of Gold Assay*

The assay silver-gold beads derived from the greater part of the converter copper now produced contain about 1 per cent. of gold. Unless the gold is obtained in coherent form in the parting operation there are almost unavoidable losses in the decanting manipulations and a resultant low gold assay. For example, when such low-grade assay beads are left in cold dilute nitric acid, nine parts water to one part nitric acid (sp. gr. 1.42), until all the silver is dissolved, the gold will be completely disintegrated. The same dilute nitric acid is the best solvent for preserving the gold in the desired state, when it is brought to the temperature of boiling water as quickly as possible after the assay bead is immersed. The higher temperature evidently increases the gold mobility.

#### *Influence of Impurities in Silver on Gold Mobility*

Silver-gold assay beads derived from silver chloride and metallic gold by the customary operations of scorification and cupellation have a silver fineness, after deduction of the gold, of 996 to 998.5 per thousand. Titrations and analyses have confirmed these figures. An analysis of 30 gm. of such beads showed them to contain 0.16 per cent. lead and 0.15 per cent. bismuth, the latter metal having concentrated in the silver from the test lead, which at the time contained approximately 0.02 per cent. of that metal. Assay beads of not less than 997 silver fineness offer no difficulty in the gold assay, but when the silver falls to 990 or lower, the gold cannot be obtained in any other form than powder, no matter what acid and heat combinations are employed.

TABLE 8.—*Assays of Copper Anode Residues*

Origin	Hot Scorification and Cupellation		Cool Scorification and Cupellation	
	Silver, Oz. per Ton	Gold, Oz. per Ton	Silver, Oz. per Ton	Gold, Oz. per Ton
In beads . . . . .	3613.12	28.030	3688.85	27.815
In slags . . . . .	55.38	0.010	56.44	0.020
In cupels . . . . .	21.64	0.045	20.93	0.025
In decantation . . . . .	...	0.075	...	0.225
Total . . . . .	3690.14	28.160	3766.22	28.085

Much difficulty was formerly encountered in obtaining concordant results for silver and gold in the assay of the same sample of copper-anode residues by the all-fire method. There were always disagreements, reading, "high silver, low gold" or "low silver, high gold." Table 8 gives the averages of 20 individual assays for each case. As the pyrometer has not been introduced in the assay muffle, it is impossible to give the terms "hot" and "cool" in degrees of temperature. By the term "hot" is meant a temperature that will keep the slag in the scorifiers thoroughly liquid and will prevent the formation of lead oxide crystals (feathers) in the cupels; by "cool" is meant a temperature that keeps the slag molten but permits the profuse formation of crystals in the cupels. Many other careful assays than those given in Table 8 showed this sample of anode residues to contain 3750 oz. of silver. The table, therefore, proves that from the operations with the higher temperature a considerable portion of the silver was not recoverable; that is, it was volatilized, while at the lower temperature impurities to the extent of 16.22 oz. were retained by the assay beads. Supposing the beads that yielded the result of 3750 oz. to be of 998 fineness, those that gave 3766 oz. should be 993.8 fine. The table shows that in the gold assay of the beads of the latter character considerable gold passes into the decantation liquid, owing to disintegration, and that it is three times as great as that from the beads obtained in the hotter operations and which, therefore, are purer. This decanted gold is not visible, ordinarily, and is not recovered in commercial work. It is best collected by the precipitation of a certain quantity of silver chloride, filtration, etc. There naturally is a gradation in the degree of the gold disintegration from its almost complete cohesion when derived from silver beads of 998 fineness to its pulverulent form when derived from beads of, perhaps, 990 fineness.

A scientific examination of the causes of these differences has not been made. It is a generally accepted theory that the whole silver-gold alloy series form solid solutions in which the two metals are, to a certain extent, in a state of continuity, their molecules remaining within spheres of mutual attraction and therefore the possibility of the cohesive property of the gold when the silver is dissolved. It may readily be imagined that impurities forming eutectic mixtures may be so interspersed in the alloy as to disrupt the continuity of the gold and to destroy its cohesive property. Or, certain impurities may form chemical compounds with the gold itself, which would no longer have the properties of the metal, but would leave it in a finely divided metallic state upon the action of the nitric acid.

## Radium

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(Colorado Meeting, September, 1918)

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PROBABLY no other metal excites as much interest, among both scientific men and the general public, as radium. This is due partly to the high cost of radium salts and partly to the peculiar properties of the element. Since radium-bearing ores were discovered in the United States, the interest of American scientific men has been stimulated and, at the present time, more radium is extracted and refined in this country than in all the rest of the world together.

## HISTORY

The property of radioactivity was discovered, partly by accident, by Henri Becquerel, the French physicist, in 1896. He was experimenting with certain fluorescent substances in order to find, if possible, a connection between fluorescence and the recently discovered X-rays. Among other chemicals which possess the property of fluorescence, he was using salts of uranium. His custom was to expose the fluorescent substance to the action of sunlight and then register the effect of possible penetrating radiation on a photographic plate protected from ordinary light. Becquerel's experiments gave positive results at once, and he at first believed that he had discovered a relation between fluorescence and X-rays. Later he exposed a plate to a uranium salt which had not been previously exposed to sunlight. To his surprise, on developing this plate, he found that he had obtained the same effect as he had previously secured when

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the uranium salt had been exposed to sunlight. The pursuit of this partly accidental discovery has given us our whole science of radioactivity. It was found that radium and its salts had the property of ionizing gases, or converting the molecules of gases into charged particles. It is this property which is used almost exclusively in identifying radioactive substances and in making quantitative determinations.

Mme. Curie, wife of the Professor of Physics in the Sorbonne, in Paris, became interested in the work of Professor Becquerel, and examined all the known elements to see whether any of them possessed properties similar to those of uranium. She found that thorium and its salts would also affect a photographic plate, without previous exposure to sunlight, and would also ionize gases. Thorium, as well as uranium, is, therefore, radioactive.

Mme. Curie found that radioactivity was an atomic property. A given weight of uranium metal had the same activity, no matter whether it was combined with chlorine, bromine, the ( $\text{SO}_4$ ) radicle or the ( $\text{NO}_3$ ) radicle. The other elements in combination with the uranium did not affect the activity in any way. She then made a study of radioactive minerals, paying especial attention to pitchblende, which is a natural uranium oxide containing traces of lead, arsenic, bismuth, and other impurities. This was furnished her by the Austrian Government from its mines at St. Joachimsthal. To her surprise, she found that a piece of pitchblende carrying a given weight of uranium had approximately four times greater activity than any pure uranium salt containing the same weight of uranium. This indicated that either her original conclusion that radioactivity is an atomic property was wrong, or the pitchblende contained another element, or elements, which were also radioactive. She proceeded to test these conclusions, and was assisted by the Austrian Government, which sent her a considerable amount of pitchblende for this purpose. The mineral was dissolved, and the different groups of elements were successively precipitated, each precipitate in turn being tested for its radioactivity. The lead group was found to be slightly active, and we know now that this was due to the presence of radium G, or radioactive lead. The copper group was also active, due to the presence of polonium, which, in many of its properties, is allied to bismuth. The activity associated with the iron group was due to actinium, which is allied to some of the rare earths. The majority of the radioactivity, however, was found to be concentrated in the barium, strontium, and calcium group. The separation of the small amount of highly active material found associated with these elements was difficult and tedious. On separating the calcium from strontium, the activity still remained with the barium; and the element, radium, was finally separated by fractional crystallization of its salts, either chloride or bromide, from the corresponding barium salts.



Mme. Curie deserves great credit for the discovery of radium and for a great deal of other scientific work she has carried out with marked success in connection with this element. But those who are not versed in the subject are likely to forget to some extent what has been accomplished by those who entered the field of radioactivity after the actual discovery of radium. On the physical side, science owes an immense debt of gratitude to Sir Ernest Rutherford. It was he who beat out the pioneer path which has given us definite radioactive theories; possessed of a keen mind and a splendid intellect, his insight has been almost uncanny. Sir J. J. Thompson, W. H. Bragg, and other physicists have added to our knowledge of this side of radioactivity; while Sir William Ramsay and Professor Soddy are responsible for a great deal that has been done on the chemical side of the subject. In this country, much has been accomplished by Boltwood, Schlundt, McCoy, and Lind.

#### WHAT IS RADIOACTIVITY ?

Radioactive substances will affect a photographic plate and will ionize gases. This is due to the fact that radium and its salts give off three types of rays, called the alpha, beta, and gamma. The alpha rays travel with a velocity of about 20,000 miles per second and are positively charged. It was early found by Rutherford that their mass was comparable to that of a helium atom; and he definitely made the statement before the proof was actually obtained that the alpha particle was a helium atom with two positive charges on it. This was afterward proved by Sir William Ramsay and Professor Soddy, who dissolved some radium chloride in water and allowed the occluded gases to run into a spectrum tube which had previously been evacuated. On allowing these gases to stand for a day or two, the spectrum of helium gradually appeared.

The beta rays consist of negatively charged electrons, with a mass of about  $\frac{1}{1600}$ th of a hydrogen atom. During radioactive changes, they are ejected with a velocity of from 100,000 to 186,000 miles per second. While the alpha particle is stopped by an ordinary sheet of note-paper, the beta particle will penetrate a thin piece of glass, but is completely stopped by a millimeter thickness of lead. All evidence points to the fact that the beta particle is similar in its properties to the electron found in a Crooks tube, and gives rise to cathode rays.

The gamma rays are not material in character but are vibrations of very short wave length in the ether. Just as the X-rays are formed in an X-ray tube by the stoppage of the cathode rays by impinging on the target, so the gamma rays are formed during radioactive changes when such changes give rise to beta rays; and these rays are expelled from the atom with the velocity of light. It is evident, therefore, that the gamma rays are practically identical with the X-rays, except that

they are of shorter wave length and penetrate matter to a much greater extent.

Anything, therefore, is radioactive which gives out alpha, beta, or gamma rays, or all of them. All radioactive changes are accompanied by at least one of these rays. The elimination is due to the explosion of the radioactive atom, such explosion taking place at a definite rate, so that in the case of radium itself one-half is completely transformed in 1690 years. In the second 1690 years, half of what is left will have been changed. In the third period of 1690 years, half of what is left at the end of the second period will have been changed—and so on. The 1690-year period is called the half-life or half-value period of radium; and it can be readily seen that in 10 times the half-life period, only about 1 per cent. of the element will remain unchanged.

### DISINTEGRATION SERIES

The manner in which radioactive elements change is shown in Table 1, giving the uranium series, and Table 2, the thorium series. Uranium 1 changes into uranium  $X_1$  with the elimination of alpha rays; uranium  $X_1$  changes into uranium  $X_2$  with the elimination of beta rays; uranium  $X_2$  changes into uranium 2 with the elimination of both beta and gamma rays—and so on down the list. It is thus plainly seen that the metal uranium is the parent of radium and, indeed, of all the radioactive elements which are found in any uranium mineral, and are shown in Table 1. This will correct what is a somewhat general impression that radium is the only radioactive element found in uranium ores in addition to uranium itself. Indeed, all of the elements of Table 1 are found in any uranium ore, and most of them can actually be separated chemically, and their physical and chemical properties identified.

It has already been stated that an alpha particle is a helium atom which has an atomic weight 4. Theoretically, therefore, whenever a radioactive atom explodes, with the elimination of an alpha particle, the resulting atom, left behind after the expulsion of the alpha particle, should have the atomic weight of the original atom minus 4, the atomic weight of the expelled helium atom. The atomic weight of radium has been determined experimentally as 226. The radium atom, during its change, loses an alpha particle with atomic weight 4 and, therefore, the residual radium emanation atom will have an atomic weight 222. By examining the fourth column of Table 1, it is seen that wherever a change occurs involving an alpha particle, the atomic weight of the resulting element is reduced by 4. As the beta particle is an electron, it has not sufficient mass to affect the resulting atomic weight.

There is some definite experimental proof that the above statements are correct. Sir William Ramsay and Professor Soddy actually deter-

TABLE 1.—*Uranium Radioactive Series\**

Uranium Series	Half-value Period	Rays	Atomic Weight
Uranium 1.....	$5 \times 10^9$ years	alpha	238
Uranium X <sub>1</sub> .....	24.6 days	beta	234
Uranium X <sub>2</sub> .....	1.15 min.	beta and gamma	234
Uranium 2.....	$2 \times 10^8$ years	alpha	234
Ionium.....	$10^5$ years	alpha	230
Radium.....	1690 years	alpha and slow beta	226
Radium emanation.....	3.86 days	alpha	222
Radium A.....	3.0 min.	alpha	218
Radium B.....	26.8 min.	beta and gamma	214
Radium C.....	19.5 min.	alpha, beta and gamma	214
Radium D.....	16.5 years	beta and gamma	210
Radium E.....	5.0 days	beta	210
Radium F.....	136 days	alpha	210
Radium G (lead).....	.....	.....	206

\* The branches of Tables 1 and 2 are omitted for the sake of simplicity.

TABLE 2.—*Thorium Radioactive Series*

Thorium Series	Half-value Period	Rays	Atomic Weight
Thorium.....	$1.5 \times 10^{10}$ years	alpha	232
Mesothorium 1.....	5.5 years	beta	228
Mesothorium 2.....	6.2 hr.	beta and gamma	228
Radiothorium.....	2 years	alpha	228
Thorium X.....	3.65 days	alpha	224
Thorium emanation.....	54 sec.	alpha	220
Thorium A.....	0.14 sec.	alpha	216
Thorium B.....	10.6 hr.	beta and gamma	212
Thorium C.....	60 min.	alpha and beta	212
Thorium D <sub>1</sub> .....	3.1 min.	beta and gamma	208
Thorium D <sub>2</sub> (lead).....	$10^8$ years	beta	208

mined the density of the radium emanation, and the figure obtained as a mean of five determinations was 223. In addition, lead is always found in uranium minerals, and the atomic weight of radium G, or the final disintegration product, according to theory, is 206. This does not coincide with the atomic weight of ordinary lead, which is 207; but some experimental work on the atomic weight of lead obtained from uranium and thorium minerals, by Professor F. W. Richards, O. Hönigschmid and Professor Soddy, has shown that the atomic weight of uranium lead is 206, while that of thorium lead is 208—an exceedingly interesting experimental confirmation of the theory. We have, therefore, actually three

forms of lead, the only difference among them being their densities, each one having exactly the same chemical and physical properties. If they were mixed by fusion, no known method could separate them; and the only way of telling one from another would be by making an actual density determination.

There is another radioactive series called the actinium series, which is very similar to the uranium series, but as it is of less importance it is not discussed in this paper.

### RADIUM ORE DEPOSITS

The two principal commercial ores of radium are pitchblende and carnotite. The former mineral has no definite composition, consisting of uranium oxides ( $\text{UO}_3$ ,  $\text{UO}_2$ ) with oxides of lead, calcium, iron, bismuth, manganese, copper, silicon, aluminum, and rare earths. Carnotite has a more definite composition, being a potassium uranyl vanadate containing small quantities of barium and calcium. The formula  $\text{K}_2\text{O} \cdot 2\text{UO}_3 \cdot \text{V}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$  expresses its composition fairly well, although not exactly. Of lesser importance are autunite, a hydrated calcium uranium phosphate  $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ , and torbernite, a hydrated copper uranium phosphate  $\text{Cu}(\text{UO}_2)_2 \cdot \text{P}_2\text{O}_8 \cdot 8\text{H}_2\text{O}$ .

#### *St. Joachimsthal*

The pitchblende deposit at St. Joachimsthal, Austria,<sup>1</sup> is in mica schist interbedded with lime schist and crystalline limestone. Toward the east and northeast the formation is gneiss. The gneiss was intruded by quartz porphyry subsequent to the deposition of the vein material. In the mica schist are fissures filled with volcanic material which cut the mineralized zone at various points and depths. The veins are usually 6 in. to 2 ft. wide, in rare cases widening out to 3 ft. The mode of mineralization varies greatly, the ores occurring in both stringers and pockets. They contain the following metals: Silver, metallic, and as argentite, polybasite, tetrahedrite, etc.; nickel, as nickelin, chloanthite, etc.; cobalt, as smaltite, bismutosmaltite, etc.; bismuth, as metallic bismuth, bismite, etc.; arsenic, as metallic arsenic and arsenopyrite; and uranium, as pitchblende and other alteration products. Galenite, zinc blende, pyrite, marcasite, and copper occur in minor quantities.

The veins show that deposition occurred in three periods: the cobalt and nickel were deposited first, then the uranium, and lastly the silver.

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<sup>1</sup> Richard Beck: "Lehre von den Erzlagertstätten." 3d Ed., 1, 408-410. Berlin, Borntraeger, 1909.

Richard Beck: "The Nature of Ore Deposits." Translation by W. H. Weed. 1st Ed., 1, 284-287. New York and London, *Engineering and Mining Journal*, 1905.

Dolomite spar is always present, and generally has a white or yellowish-white color, but changes to a brownish-red hue where pitchblende begins to appear, and is a dirty gray where it is actually in contact with the ore. Deep-blue fluorspar is always present.

The mines at Joachimsthal have been worked since 1517. In 1545 the production of silver ores declined considerably, but since then the deposits have been mined for bismuth and cobalt. During the last 25 years the mines have been worked for uranium.

### *Saxony*

In the vicinity of Annaburg, on the Saxony side of the Erzgebirge, the silver-cobalt veins resemble those at Joachimsthal. At Johanngeorgenstadt, the veins contain tin and silver-cobalt ores. Where dolomite spar is found, the silver-cobalt ores contain pitchblende, as at Annaberg. In the Gottessegen mine the pitchblende occurs in the spar in pieces 2 to 3 in. in diameter. These mines are worked principally for bismuth ore, but also for cobalt and nickel.

In the cobalt-bismuth mines of Schneeberg, are found bismutite and various minerals of nickel, silver, and arsenic. There is also some pitchblende, uranocalcite, uranospinite, galenite, zinc blende, etc.

### *Cornwall*

Pitchblende has also been found in Cornwall, England, in the tin region. As at Joachimsthal and Johanngeorgenstadt, the mineral is found associated with nickel-cobalt veins, although only part of the veins are highly argentiferous. Even though these veins are closely connected with the tin veins, they apparently are not of the same age as the latter, but belong to the same general period of mineralization. According to Ussher, Barrow and MacAllister,<sup>2</sup> the most important uranium producers are the South Terras mine, the Carharrack, Dolcoath, Wheal Unity, Wheal Gorland, Wheal Lovell, and Trenwith. The South Terras mine is situated in the valley of the Fal, southwest of St. Austell. The country rock is slate, intruded by greenstone and granite porphyry dikes.

### *Pitchblende in the United States*

Pitchblende has been found in the following localities in the United States: Feldspar quarry, at Middletown, Conn., in large octahedrons; in Hall's quarry, at Glastonbury, Branchville, Conn., in a pegmatite vein and usually embedded in albite; at Marietta, S. C.; in the Baringer

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<sup>2</sup> The Geology of the Country Around Bodmin and St. Austell. *Memoirs*, Geological Survey of England and Wales (1909), 157.

Hill district, Llano County, Texas; in the Bald Mountain district, Black Hills, S. Dak.; in Mitchell County, N. C.; and in Gilpin County, Colorado. The latter district is the only one of commercial importance.

All of the Gilpin County mines, with one exception, are found on or near Quartz Hill, a few miles from Central City. There are five that have produced pitchblende in quantity: the Kirk, Wood, German, Belcher, and Calhoun. The Kirk, Belcher and German mines are close together on Quartz Hill, the Wood and the Calhoun being in the valley below.

These mines, until recently, have been worked mainly for gold. In this district, gneiss and crystalline schist predominate, with intrusive andesitic dikes and occasionally acid granitic dikes. The rock containing the pitchblende, galena, sphalerite, etc., is a fine-grained aplitic granite which probably once contained an appreciable amount of biotite. The ore deposits are of two general types, one containing pitchblende with pyrite, sphalerite and galena, and sometimes marcasite; the other type contains pyrite, chalcopyrite, sphalerite and galena, with some gold and silver. Generally speaking, the two types are not associated, so that the miner has a choice of mining either for pitchblende, or for gold.

The Kirk mine has probably been the most important producer of the five mentioned although reliable data on the output of pitchblende from this mine, up to a few years ago, has been difficult to obtain. During the last 12 years, about 20 tons of ore, with an average content of 35 per cent.  $U_3O_8$ , and over 100 tons with a content of 3 to 4 per cent.  $U_3O_8$ , have been mined. The mine has been shut down for some time. More recent operations of the German and Belcher mines produced 120 tons of low-grade ore, averaging about 1 per cent.  $U_3O_8$ , and 6 tons of high grade. Smaller quantities of ore have been produced at various times from these mines and from the Wood and the Calhoun.

### *Australia*

Uranium ores are found in certain localities in Australia. One of these deposits is 80 miles east of Farina, a railroad station on the Great Northern line in South Australia, and lies between Mount Painter and Mount Pitt. H. L. Y. Brown<sup>3</sup> states that the rocks of the district consist of coarse and fine feldspathic, siliceous, and micaceous granite, gneiss, quartzite and mica schist. The rocks are contorted in places and penetrated by dikes of coarse, pink-colored eruptive granite.

Two of the prospect pits are on outcrops of iron oxide with cellular quartz and gossan, the whole having the appearance of an irregular lode. The uranium minerals, torbernite and autunite, are disseminated through the ore, and are also crystallized on the walls of the fissures and

<sup>3</sup> Occurrence of Uranium Ores and other Rare Minerals near Mount Painter, in the Flinders Range of South Australia. South Australia, Mines Dept., 1911.

cavities in it. Uranophane and gummite occur sparingly; fergusonite and some monazite are also present. Another uranium deposit lies southeast of the one just described, about 20 miles southeast of Olary, on the railroad line from Petersburg to Broken Hill, South Australia. The ore occurs as a yellow and greenish yellow incrustation and powder on the faces, joints and cavities of a lode, which consists of titaniferous magnetite, magnetite, etc., and quartz in association with black mica.

### *Portugal*

Autunite is found in commercial quantities in Portugal in the district between the towns of Guarda and Sabugal. An excellent description is given by Segaud and Humery.<sup>4</sup> Apart from the uranium, the rocks of the region are much mineralized, showing deposits of galena, arsenopyrite, chalcopyrite, tungsten and cassiterite.

All of the deposits referred to above are important and have been developed as commercial sources of radium; in fact, until about 7 years ago, they were the only sources from which radium ores were obtained.

### *Carnotite Deposits of Colorado and Utah*

About 1910, the carnotite deposits of southwestern Colorado and eastern Utah began to receive attention. They were known as far back as 1881, but the composition of the ore was unknown until 1897. In 1899, an analysis showed that the ore not only contained uranium, but was a potassium uranyl vanadate.

In 1900, a small plant was erected in the McIntyre district, south of the Paradox Valley, Colo., for the extraction and recovery of uranium oxide from carnotite ore. Only moderate success was achieved, and the plant was shortly shut down. Operations were also started by other concerns, notably the Western Refining Co. and the Dolores Refining Co.; these plants extracted uranium and vanadium only. None of these operations was of importance, and it was not until 1909 to 1910 that any interest was shown in the carnotite deposits as a source of radium. At that time, the ore was almost exclusively shipped abroad.

In the fall of 1912, representatives of the U. S. Bureau of Mines made a thorough survey of the carnotite fields and announced the fact<sup>5</sup> that the carnotite deposits of Colorado and Utah constituted by far the largest source of radium-bearing ores in the world. Developments since that time have proved this statement to be correct, as the larger part of the radium that has been produced in the world has been derived from American carnotite ore.

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<sup>4</sup> Segaud et Humery: Les Gisements d'Uranium du Portugal. *Annales des Mines, Mémoires*, Ser. 11 (1913) 3, 111-118.

<sup>5</sup> Richard B. Moore and Karl L. Kithil: A Preliminary Report on Uranium, Radium and Vanadium. U. S. Bureau of Mines, *Bulletin* No. 70 (1913).

The deposits are found mainly in Dolores, San Miguel, and Montrose Counties, Colorado, and extend over a belt about 60 miles long by 20 miles wide. The ore is also found to the west of the La Sal Mountains in Utah, and along the San Rafael Swell, southwest of Green River, Utah. Small patches of ore are found scattered between these points and extend as far north as Meeker, Colorado.

The most usual ore is a sandstone so impregnated with yellow carnotite that the color is decidedly noticeable, and contains small kidneys of brown sandy clay. The kidneys constitute a considerable part of some of the ore; in many cases they are thinly scattered through the sandstone. Although ore of the character mentioned is widely distributed in the Paradox and adjacent districts, and constitutes a large part of the ore shipped, it is by no means the only ore of commercial importance. Indeed, the variety of the types of ore here and in Utah is one of the interesting features of the uranium and vanadium deposits. There are dark blue, brown, and black vanadium ores, the dark blue ores being lustrous when first mined and usually carrying uranium; high-grade carnotite in vug holes so soft that it can be molded in the fingers; and the same kind of ore crystallized with gypsum. Red calcium vanadate is found alone and mixed with carnotite. The ores of the Paradox district differ in many respects from those in Utah, but chiefly in carrying larger proportions of carnotite, and as a rule are more yellow. Not only do they carry more uranium but also, on the average, more vanadium, although individual shipments from Utah, particularly from Temple Mountain, might seem higher in vanadium than the average ore from the Paradox district.

The deposits are invariably pockets, many of which, however, are of considerable size. A few individual claims have produced as high as 500 tons of shipping ore, which, however, is exceptional. The ore is found in a light-colored sandstone overlain in places with shale and conglomerate; according to Hillebrand and Ransome, this is the McElmo formation.<sup>6</sup>

#### METALLURGICAL TREATMENT

The average commercial ore from which radium is extracted contains from 5 to 10 milligrams of radium element per ton of ore. Allowing for losses in extraction and recovery, it takes about 5000 or 6000 tons of ore to give an ounce of radium element. It can be seen, therefore, that the metallurgical processes involved in the extraction of radium are entirely different from those connected with any other element. There are two general steps which must be carried out: first, to obtain

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<sup>6</sup> W. F. Hillebrand and F. L. Ransome: On Carnotite and Associated Vanadiferous Minerals in Western Colorado. U. S. Geological Survey, *Bulletin* No. 262 (1905), 11.



a radium concentrate from the ore; and second, the re-treatment of this concentrate in order to extract a high-grade product. The first step is necessarily carried out on a large scale, while the major part of the second step is done in the laboratory.

The different methods of treating radium-bearing ores to obtain a concentrate may be classed under three general heads: (1) an alkaline leach, followed by an acid leach; (2) fusing the ore with some material that will disintegrate it and make the extraction of the valuable contents possible; (3) an acid leach.

### 1. *Alkaline Leach Methods*

It is probable that some of the early experimental work on extracting radium from carnotite ore involved boiling the ore with a solution of sodium-carbonate, thus getting rid of the uranium and vanadium, which go into solution. Since radium has properties similar to those of barium, any radium in the ore would be converted into radium carbonate, and on treating the residue with dilute c. p. hydrochloric acid, the radium would be dissolved with any other acid-soluble products.

The radium concentrate always obtained is radium-barium sulfate. By adding barium chloride and sulfuric acid, or sodium sulfate, to the slightly acid solution carrying the radium, barium sulfate is formed in the solution and drags down radium sulfate with it. The radium is almost always precipitated in this manner in a liquor sufficient in volume to hold it actually in solution. Undoubtedly adsorption has something to do with the precipitation of the radium along with the barium sulfate, but this does not fully explain the small losses that accompany such precipitation. The term "pseudo-isotopy" has been given to this property by Dr. S. C. Lind.<sup>7</sup>

The general principles outlined above are included in the Haynes-Engle process, which involves boiling the ore with an alkaline carbonate solution. The object of this process was to recover uranium and vanadium only, and did not attempt to obtain the radium in any form. A patent taken out by Warren F. Bleeker involved the extra step which the Haynes-Engle process did not cover, namely, the leaching of the residues with hydrochloric acid in order to obtain the radium in solution, after the ore had been boiled with an alkaline carbonate solution.

The alkaline leach method has many advantages and some disadvantages. It separates the uranium and vanadium from the ore during the first stage of the process. It eliminates sulfates by converting the metallic sulfates in the ore into metallic carbonates and soluble sulfates, which go into the filtrate with the uranium and vanadium. The radium,

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<sup>7</sup> S. C. Lind, J. E. Underwood, and C. F. Whittemore: The Solubility of Pure Radium Sulfate. *Journal, American Chemical Society* (March, 1918) **40**, 465-472.

therefore, is left behind in the residue as carbonate, practically free from sulfates. This prevents the re-precipitation of the radium as sulfate, on treating with acid, until after the acid solution is filtered from the tailings. On the other hand, it has the following disadvantages: It converts some of the silica in the ore into sodium silicate, which makes filtration very difficult—in fact, most of the filtering and washing has to be done by decantation. It is difficult to treat concentrates obtained by this process, as these are almost invariably of very fine mesh, which adds to the filtration difficulties. The treatment with alkaline carbonate converts most of the iron and a good part of the aluminum in the ore into an acid-soluble form, so that the acid consumption is high. The method, however, can be used with success for the treatment of certain uranium ores, particularly carnotite, autunite, and torbernite.

## 2. *Fusion Methods*

The first method used for the extraction of radium was fusing pitchblende ores with sodium sulfate. This method was originally used by the Austrian Government in connection with the treatment of pitchblende ores from its mines in St. Joachimsthal. By this fusion, the uranium in the ore is changed to sodium uranate, which can be dissolved from the insoluble residue, after leaching out the excess of sodium sulfate with water, by dilute sulfuric acid. The radium remains behind with the residue and, before the discovery of radium, was discarded. Afterward, the residue was boiled with sodium carbonate, which converted a portion of the radium sulfate into radium carbonate, and this was leached out with dilute hydrochloric acid. After repeating this process several times, practically all of the radium was leached from the residue, and the acid leaches were combined. Barium sulfate was then precipitated in the acid solution in the ordinary manner, and in this way the radium was obtained as a concentrate with the barium sulfate.

A somewhat similar treatment is given to the ores from Olary, South Australia, by the Radcliffe process. The main uranium mineral is carnotite, but this is associated with considerable quantities of ilmenite and other rare-earth minerals that are not found in American carnotite. The concentrates are mixed with three times their weight of salt-cake, and fused in a reverberatory furnace; the fused product is then crushed and agitated in wooden vats, with water. By suitable adjustments it is possible to separate on the bottom of the vats a considerable amount of comparatively coarse material that is almost free from radium and uranium. The turbid overflow carries in suspension the radium, lead, and barium, as sulfates, together with a considerable amount of finely divided silica. The overflow is pumped to large lead-lined tanks and allowed to stand all night. This is nothing but a sliming process and has the advantage that the radium in the form of sulfate always remains with

the fine material. The slimes settle completely in 12 hr. and are collected periodically and treated for the recovery of radium.

The process of Schlundt, which is being used by one company in the United States in connection with carnotite, is very similar to the Radcliffe process. The ore is fused with acid sodium sulfate, leached and washed with water to extract the uranium, vanadium, and other soluble products, and the residue is slimed in order to obtain a crude concentration of the radium, which stays with the fine material.

The U. S. Bureau of Mines has found that if a radium-barium sulfate high in silica is fused with caustic soda containing a small quantity of sodium carbonate, the silica can be easily washed out as sodium silicate, while the barium and radium remain behind as radium-barium carbonates, which can be readily dissolved in hydrochloric acid. Usually the commercial caustic soda contains enough carbonate without the addition of an extra amount. This method has been applied commercially to the treatment of crude concentrates, such as are obtained by the Schlundt process.

One firm has used a sodium carbonate fusion. The ore is fused with soda ash, usually about  $2\frac{1}{2}$  times the weight of the ore being required. This is done in a reverberatory furnace, lined with magnesite brick, and the fused mass is run directly into vats, in which it is leached. The silica is thus converted into sodium silicate and so passes into solution, together with the uranium and vanadium. The iron, calcium, barium, radium, etc., remain as the insoluble residue, which is washed in filter presses. This material is then treated with dilute sulfate-free hydrochloric acid, which dissolves the carbonates, and the radium and barium are precipitated by the addition of the requisite amount of sulfuric acid, or sodium sulfate. The whole is allowed to stand in settling tanks and the clear liquid is drawn off, leaving the barium-radium sulfates, mixed with a considerable amount of silica and other impurities, as a sludge at the bottom of the tank. This is taken off without previous filtration, and dried, forming a crude radium-barium sulfate, which is then refined.

The main advantages of this method are that it will treat an ore containing considerable quantities of sulfates, as the process removes the sulfates as soluble sodium sulfate in a similar manner to the alkaline leach method. In addition, it is applicable to the treatment of concentrates, as fineness of material is really an advantage instead of a disadvantage, as it does not involve any "sliming." The main disadvantage is the cost, both for the chemicals and the labor required. The concentrate obtained is also low-grade, which involves additional refining costs.

### 3. Acid Leach Methods

Leaching with hydrochloric acid gives an excellent extraction, provided the ore is comparatively free from sulfates. As a considerable propor-

tion of such ores contain traces of gypsum, it is a method which must be used on selected ores. A hydrochloric acid leach has been used successfully on Cornish pitchblende, which is practically free from pyrite, but is not applicable in any way to American pitchblende, which contains considerable quantities of pyrite. It has also been used on Portuguese autunite. In general, the hydrochloric method is applicable to certain ores, but has a limited use.

Leaching with nitric acid has been used more successfully. This is the method originated by the U. S. Bureau of Mines<sup>8</sup> and used in the plant of the National Radium Institute. As is generally known, barium sulfate is much more soluble in nitric acid than in hydrochloric acid, and this is especially true when the nitric acid is concentrated and hot. For this reason, ores containing sulfates can be successfully treated by a nitric acid leach, and the method has been successfully applied to ores carrying as much as 1 per cent. gypsum. On ores carrying small quantities of sulfates, the extraction obtained is very high, frequently going, on a commercial scale, to 93 and even to 95 per cent. Boiling, 40-per cent. nitric acid is used, and filtration is obtained on either a vacuum or a pressure filter. Filtration must be done rapidly, because, as the acid cools, the radium has a tendency to precipitate out, especially in the presence of sulfates. During the process of recovering uranium and vanadium by this method, practically nothing but sodium hydroxide and sodium carbonate are added to the nitric acid. Consequently, sodium nitrate is obtained as the final product after the precipitation of the radium, uranium, and vanadium; and this sodium nitrate is recovered by evaporation and crystallization and used over again for the manufacture of nitric acid in the ordinary way. The average losses of nitric acid were about 15 per cent., so that 85 per cent. of the acid used was recovered as sodium nitrate. This actually reduced the cost of the nitric acid below that of hydrochloric, and this, together with the high extraction of the radium obtained, was largely responsible for the low cost of the recovered radium and the success of the method.

The process cannot treat successfully ores carrying as large quantities of gypsum as can be treated by some of the other methods, such as the sodium carbonate fusion, but it is applicable to a very large percentage of the carnotite ore produced. Difficulty was experienced in treating concentrates, most of which are below 150 mesh. As already stated, it has been necessary to filter rapidly in order to get a good extraction; and the problem of filtering a 40-per cent., boiling nitric solution, containing fine material in suspension, was found to be difficult. It was finally overcome by the development and use of a pressure filter instead of a

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<sup>8</sup> Charles L. Parsons, R. B. Moore, S. C. Lind, and O. C. Schaefer: Extraction and Recovery of Radium, Uranium, and Vanadium from Carnotite. U. S. Bureau of Mines, *Bulletin* No. 104 (1915).

suction filter; this consisted of a steel shell containing an earthenware filter set in concrete. The earthenware filter had at its bottom, as filtering medium, a plate of either filtros or alundum. The steel top was made tight with swing bolts, and a pressure of 100 lb. was applied.

### *Refining*

The radium-barium sulfate concentrate obtained in a satisfactory metallurgical process should carry about 1 mg. of radium element per kilogram of concentrate, a ratio of one to a million. The next step is to get this concentrate back into solution. The most successful method is to reduce the sulfate with about one-sixth its weight of pulverized charcoal in a furnace at a temperature from 800° to 900° C. In the presence of silica, the reduction takes place slowly and is not complete; but if the sulfate does not contain more than from 5 to 10 per cent. silica, the reduction may reach 90 per cent. or more. The radium-barium sulfides are then dissolved in sulfate-free hydrochloric acid, and filtered. From this stage on, the refining process is simply one of fractional crystallization. Radium chloride and bromide are less soluble than the corresponding barium salts; the crystals first obtained on evaporation are therefore richer in radium than the mother liquor from which they are obtained. The difference in solubility of the bromides is considerably greater than that of chlorides; at some stage in the process, all of the chloride salts are converted into bromides, after which fractionation is continued as bromide, in hydrobromic acid solution. The factor of separation is higher in acid than in neutral solution; it is customary, therefore, to fractionate both chlorides and bromides in acid solution. Full details of the methods of fractionation are given in U. S. Bureau of Mines *Bulletin* No. 104.<sup>9</sup>

### METHODS OF ORE CONCENTRATION

Both wet and dry methods of concentration may be used for carnotite ores. The wet method simply involves crushing the ore and agitating with water in a manner that will best separate the fine-grained particles of carnotite from the coarser silica particles. After allowing to settle, the fine material can be slimed off and run into settling tanks, while the coarser tailings are re-treated with water. The only successful method of dry concentration that has so far been used has involved the Raymond pulverizer and dust collecting system. The National Radium Institute built a small plant near the claims it was working in Long Park, Colo., and gave the method a thorough testing, with excellent results.<sup>10</sup> The

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<sup>9</sup> *Op. cit.*

<sup>10</sup> Karl L. Kithil and John A. Davis: Mining and Concentration of Carnotite Ores. U. S. Bureau of Mines, *Bulletin* No. 103 (1917).

capacity of the mill was a little over one ton of milling ore an hour; and the average output was 365 lb. of concentrate an hour. The milling ore had an average content of 0.85 per cent.  $U_3O_8$ ; the average of all concentrates was 2.92 per cent.  $U_3O_8$ ; the tailings averaged 0.3 per cent.; the ratio of concentration was about 6 to 1. As much as 63.7 per cent. of the carnotite contained in the milling ore was extracted, and 60 per cent. of all the carnotite in this ore was actually recovered in the concentrates.

#### FUTURE SUPPLY OF ORE

It is difficult to estimate the exact amount of radium in existence at the present time; probably it is somewhere around 3 oz. of radium element. On account of the great scientific interest attached to radium, and on account of its use in the war and in medicine, the permanency of the ore supply is an important question. Considerably more than half the amount of radium now in existence has come from Colorado and Utah carnotite ores. Six years ago, the engineers of the Bureau of Mines estimated that, at the current rate of production, the deposits might last, commercially, 10 or 12 years. At the present time, it is very difficult to obtain ore. Most of the deposits are owned by five operating radium companies. The production has increased very greatly during the war; and the author is very doubtful whether we can depend upon our carnotite deposits to yield commercial quantities of ore for more than 6 or 7 years longer. It is his judgment that the fields will not produce more than 100 additional grams of radium element at the most—if that much. This would about double the world's present supply; but, on account of the large use of radium in cancer treatment, such an amount, although large scientifically, would be small in proportion to the probable demand.

TABLE 3.—*Radium Production from American Carnotite*

Year	Tons $U_3O_8$	Tons U	Grams Ra Contained	Grams Ra Recovered, (75% Basis)
1912	28.8	24.4	7.4	5.5
1913	41.0	34.8	10.5	7.9
1914	87.2	74.0	22.5	16.9
1915	23.5	19.9	6.0	4.5
1916 (a)				
1917	70.0	59.3	18.0	13.5

(a) Official statistics are not available for 1916.

#### USES OF RADIUM

The use of radium in cancer research has extended over a period of several years. Some very remarkable results have been obtained, especially recently in the treatment of certain forms of cancer. A large

number of permanent cures have been effected; and whereas the medical fraternity was skeptical at first, a great deal of the opposition has been overcome, and several of the largest and most prominent hospitals in the country are now supplied with considerable quantities of radium for cancer treatment. A word of warning must be given in this connection. The successful use of radioactive waters and low-grade radium preparations is doubtful and should be carefully distinguished from the successful results obtained with considerable quantities of high-grade radium salts. Naturally, there have been some attempts to foist on the public, at a high price, low-grade preparations, the value of which is, to say the least, very uncertain.

The chief technical use for radium is in connection with permanently luminous paint. If radium salts are mixed with phosphorescent zinc sulfide, a product is obtained which is permanently luminous in the dark without previous exposure to light. The zinc sulfide must be specially prepared and usually contains traces of impurities, such as copper, manganese, etc. Just what relation exists between these impurities and the peculiar properties of the zinc sulfide is not known, although experimental work is now being done to determine it. The luminous paint usually carries from 0.1 to 0.25 mg. of radium element to 1 gm. of zinc sulfide, depending upon the particular use of the paint.

This material is now being largely used on the faces of watches and clocks; to coat electric light push-buttons and the chains attached to electric globes; and for various other purposes. It is a great pity that our supply of radium is being disseminated in this manner. But as the physicians and surgeons of the country are not purchasing enough radium to make the industry a financial success, it is natural that the manufacturers should take other means of creating a demand. The day is not far distant, in the judgment of the writer, when we shall greatly regret the radium that has been lost in this way.

Radium has a most decided war use at the present time; nine instruments used on airplanes have dials made luminous with radium paint; it is employed in the same manner for compasses and gunsights. The efficiency of night firing, with both machine guns and artillery, has been greatly increased by the use of these luminous sights. Other uses cannot be specified, at the present time, in a public paper.

#### MESOTHORIUM AS A SUBSTITUTE FOR RADIUM

One way of preventing the dissemination and loss of radium is to provide a substitute. Mesothorium (see Table 2) is an excellent substitute in many ways. Its half-life period is much shorter than that of radium. When first prepared, it gradually increases in activity, comes to a maximum, and then begins to lose its activity. After "ripening"

for about a year after being prepared, it can be used for luminous paint just as efficiently as radium. Its usefulness for such purpose will last for 4 or 5 years, which is as long as is required for cheap watches, push-buttons, etc. Mesothorium can be obtained as a byproduct in the treatment of monazite sand for the manufacture of thorium nitrate used in incandescent mantles. During the last year, the U. S. Bureau of Mines has been experimenting along these lines and has developed a process which is being put into the largest thorium plant in the country at the present time, and it is hoped that before long mesothorium can be substituted for some of the radium that is now being used in luminous products. Mesothorium can also be used for cancer treatment, although its short life makes it much less desirable for this purpose than radium.

## DISCUSSION

W. A. SCHLESINGER,\* Denver, Colo.—Two problems are of great interest to the radium manufacturer at the present time: The first is to perfect a process, more efficient and more economical, which is capable of treating a greater variety of ores. Practically all the radium now manufactured in the United States is extracted from carnotite ores and these vary a great deal according to their location. The amount of sulfates contained in the ore is of particular importance, and any straight acid-leaching method, such as developed by the Bureau of Mines, becomes prohibitive when the ore contains more than 0.5 per cent. of sulfates, as the sulfates go into solution and naturally precipitate the radium as insoluble radium sulfate. Since the amount of ore obtained from any one claim is usually comparatively small, it is necessary to work a number of claims in different localities, and a process, in order to be commercially satisfactory, should handle all of these ores. In other words, it would be entirely unprofitable for radium manufacturers to employ any process which cannot treat any but sulfate-free ores. He must deal with carnotite containing appreciable amounts of sulfates, and must be able to work this in with the other ores. Carbonaceous ores from certain sections in Utah, which are known to contain as much as 10 per cent. and more of volatile matters, are prone to a nitric acid-leaching process.

The second problem relates to the treatment of concentrates. There is no doubt that a very large amount of low-grade ore, containing less than 0.75 per cent. uranium oxide, is available. It has been demonstrated that these ores can be concentrated, but **there** is still room for research work to develop a process that will efficiently **handle** such concentrates. The straight acid-leaching method, if used with concentrates, is con-

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\*The Radium Company of Colorado, Inc. (Formerly The Schlesinger Radium Co.)



fronted by difficulties in filtering. The concentrates are naturally very fine, and when leached with acid will easily clog the filtering medium.

It was suggested that, by mixing concentrates with straight oreground to 20 mesh, one could overcome the filtering difficulties, with a straight acid-leaching method on sulfate-free ores, by the use of pressure filters, and obtain an extraction of approximately 75 per cent. If, however, this method gives an extraction of 95 per cent. on the straight ore alone, and if by mixing with 25 per cent. of concentrates the combined extraction is reduced to 75 per cent., no advantage is gained by this procedure.

A considerable amount of radium has lately been used in the manufacture of self-luminous compounds, consisting of specially prepared zinc sulfide mixed with varying amounts of radium. The powder is then, with the help of a suitable varnish, made into a paint and applied with a brush. The character of the varnish varies according to the use to which one wishes to put the paint, and whether or not the paint has to withstand the action of alcohol, glycerine, or water. We have applied self-luminous radium preparations to over half a million Government instruments since the outbreak of the war. Units for luminosity measurements have also been developed, and the luminosity of these paints is now being measured in micro-lamberts, which are a fraction of a candle power.

The suggested use of mesothorium is very interesting, but little has been published and few experiments have been made in this country to show whether mesothorium is as efficient as radium when mixed with zinc sulfide for luminous paint. Experimental data should be collected to show the rate of deterioration of zinc sulfide when mixed with mesothorium.

I do not think a shortage of radium will occur for years to come, especially in view of the low-grade deposits. The amount of radium used for luminous paints, even now, is comparatively small; probably 500 milligrams is being used every month for the manufacture of luminous paint for war purposes in this country.

R. B. MOORE.—It would, of course, be an excellent thing if we could have one process, as suggested by Dr. Schlesinger, that would be equally efficient in connection with all types of carnotite ore. This, however, is almost too much to ask as the radium ores are just as varied in character as are copper ores, and we do not expect any process for the recovery of copper to be equally efficient on all types of ore.

I have been asked to say a few words about Dr. Douglas in his connection with radium. The radium I have showed you was some which was produced by the National Radium Institute, in which Dr. Douglas was greatly interested; in fact, he was one of those who financed that organization. He did this mainly for two reasons: first, as a philanthropic object for the alleviation of suffering from cancer, and secondly, because of his interest in scientific and technical progress of all kinds.

At that time the extraction of radium from ores had been only slightly investigated; practically nothing was known about it in this country. All of the radium he obtained as his share in the operations went to the Memorial Hospital, in New York, and is still there as a gift to the Hospital. Dr. Douglas, therefore, accomplished the two objects he had in mind—he helped to make possible a study of the technical methods of extraction and at the same time gave his share of the radium obtained for research work in cancer treatment.

### Biographical Notice of James Douglas

BY ROSSITER W. RAYMOND

It is scarcely necessary to augment or amend the "Appreciation" of Dr. Douglas, from the pen of Dr. Albert A. Ledoux, which appeared in January, 1916, in *Bulletin* No. 109 of the Institute. The author of that admirable summary enjoyed the great privilege of expressing while Dr. Douglas was yet among us (though already marked for death) the love and esteem with which we all regarded him. But now that his earthly life has ended, the historian must still be the eulogist. No line need be added or deleted in the portrait of him already drawn; it has only to be accepted now as a true likeness, and hung in the gallery of the immortals, illuminated by the crowning glory of another world.

James Douglas was born at Quebec, Canada, November 4, 1837. His father, James Douglas, was a distinguished physician and surgeon, known in many lands, especially in the Orient, and famous in his own country for his philanthropy as well as his skill, having established and directed for many years the first retreat for the insane in the Dominion of Canada.

One of the latest literary labors of the son was the editing and publishing of his father's journal and reminiscences—a fascinating volume, the review of which, though a tempting task, I must here forego. Nearly fifty years ago, I had the great pleasure of spending an hour with the elder Douglas, who guided me through his collection of Egyptian and Asiatic treasures. I might almost say souvenirs; so many of them were connected with personal experiences and exalted personages. The veteran's memory had already begun to fail with age, but his vivacity and imagination glowed all the more brightly; and his reminiscences of travel and adventure were embroidered with Oriental magnificence. I felt, after that hour, as if I had visited a stately, half-ruined edifice, overgrown with vines and flowers.

The father's genius, adventurous spirit and generous philanthropy descended to his versatile, yet practical son. Like other men of such temperament, James Douglas tried many things before circumstances beyond his own control forced him into the line of his principal lifework. At the age of 18, he was sent to the University of Edinburgh, where he studied for two years. Returning to Canada, he entered Queen's University, at Kingston, Ontario, where he was graduated as Bachelor of Arts in 1858. Subsequently he studied medicine at Laval University, Quebec;

and it was doubtless during this period that he assisted his father in the management of the Quebec lunatic asylum; and also traveled extensively with him in Europe and the East. In connection with his study of medicine at Laval University he became interested in chemistry, which he afterward taught for several years at Morrin College, Quebec. No doubt his excursion in this direction was initiated by his acquaintance with a man who was destined to have a decisive influence upon his future career, namely, Thomas Sterry Hunt, at that time about 33 years old, who was lecturing on chemistry in the French language at Laval. Hunt's prodigious intellectual activity, keen insight into the facts and laws of nature, and fierce enthusiasm in the pursuit of scientific truth (qualities evident enough in later years, but doubtless supereminent in his youthful prime) must have affected profoundly a mind like that of Douglas. The two became close friends and in after years business partners. Yet meanwhile their paths were widely sundered. Hunt continued his brilliant career as chemist and geologist, on the Canada Geological Survey, in the faculty of McGill University, on the juries of successive international expositions, as the recipient of sundry honorary degrees and decorations, as prolific author of notable scientific papers, and finally as Professor of Geology in the Massachusetts Institute of Technology, and one of the most active promoters and officers of the American Institute of Mining Engineers. Douglas, meanwhile, returned to Edinburgh, to continue the study of medicine—choosing, however, this time the more scientific branch of surgery—and pursuing as an avocation at the same time a course on theology, which he carried so far as to receive a license to preach. It is permissible to conjecture that he prophetically foresaw the type of spiritual leader who ministers to both soul and body—a type more fully developed nowadays in the medical missionary, and furnishing for Douglas a welcome compromise, or rather combination, of activities, satisfying at once his love of science and his love of men. But his professional plans were cut short by the pecuniary embarrassments of his father, who had made unfortunate investments in mining schemes. To assist him, the younger Douglas returned to Canada, about 1871.

The investments referred to were in the Harvey Hill copper mines in Quebec, the 2-per cent. copper ores of which could not be economically treated at that time by any known process. Douglas was doubtless familiar with the researches of Sterry Hunt on the reactions between cupric oxide, sulfur dioxide, etc., and had recourse to his old friend in this emergency. Together they worked out the famous Hunt and Douglas process, the original form of which was described by Hunt as follows:<sup>1</sup>

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<sup>1</sup> T. Sterry Hunt: Remarks on the Hunt and Douglas Copper Process. *Trans.* (1871-73) 1, 258.

The essential principle of this new process \* \* \* \* is the dissolving of the oxides of copper by a hot solution of protochloride of iron and common salt. In the action which takes place, the protochloride of iron is converted into peroxide, while the oxides of copper are changed to protochloride and dichloride, the latter of which, though insoluble in water, is readily soluble in a hot, strong brine. From the solution thus obtained, metallic iron throws down the copper in a metallic state, regenerating the protochloride of iron, which is then ready for the treatment of a fresh portion of oxidized copper ore.

The obvious ingenuity and beauty of this process made it very attractive to metallurgists; and for a time it was believed that the treatment of 2-per cent. copper ores had been made economically practicable in this way. It would be easy but tedious to enumerate here the many practical and commercial difficulties which have proved, so far, insurmountable by such methods. In 1893, Douglas himself wrote:<sup>2</sup>

There has been, however, but little patronage extended to wet copper-methods, mainly because we do not possess, within accessible reach of the chemical centers, any large bodies of cupriferous pyrites, whose residues, after the extraction of sulphur and copper, would possess value as an iron-ore. The treatment of the low-grade oxidized ores of the Southwest is awaiting realization. In the past, various attempts have been made to employ old and new wet methods; but none has proved commercially successful, nor has any survived until to-day.

In 1875, Mr. Douglas came to the United States as Superintendent of the Chemical Copper Co., of Phoenixville, Pa., which treated copper ores and pyritic cinders, and also melted and refined base metal. A variety of the Hunt and Douglas process was employed for extraction. It was a small plant, and ill-supplied with capital. I remember examining it while Douglas was in charge, and admiring the courage with which he struggled against technical difficulties, and the ingenuity with which he devised substitutes for expensive apparatus. It was an up-hill business, and after some years of strenuous endeavor, the plant was destroyed by fire. But the discipline of the long contest had made a strong man of Douglas, while his business relations had brought him into contact with many who could not fail to be impressed by his ability and integrity. For some years after the failure of the Phoenixville enterprise, he was without fixed employment, though he did some important consulting work, visiting Montana, Colorado and other mining districts. Concerning the stroke of well earned "good luck" which placed him on the straight road to fame and fortune, I cannot do better than quote the story as told by Dr. Ledoux:

An accident brought him into contact with the old metal house of Phelps, Dodge & Co. When the Copper Queen mine was opened by Martin and Reilly, the first

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<sup>2</sup> James Douglas: Summary of American Improvements and Inventions in Ore-crushing and Concentration, and in the Metallurgy of Copper, Lead, Gold, Silver, Nickel, Aluminum, Zinc, Mercury, Antimony and Tin. *Trans.* (1893) 22, 321 (Chicago meeting, 1893, being part of the International Engineering Congress).

carloads of copper bars were sent to Phoenixville to be refined by Dr. Douglas's works. He had been introduced to Mr. William E. Dodge and had been retained to report on the Detroit Copper Company's mines in Arizona. This firm was conservative in the extreme and, while very large sellers of metals, had but recently entered into the mining field, considering mining somewhat of a gambling venture. Urged by an acquaintance, they had taken an option on the former Copper Queen—the original of the name—in Arizona, and engaged Dr. Douglas to examine it. They agreed to pay his expenses and to furnish him with a certain sum of money with which to test the property, promising that if they took it over on his recommendation, they would place the management in his hands and give him an interest.

The world knows to what great heights Phelps, Dodge & Co. have attained in the mining business. Dr. Douglas, upon the incorporation of the firm, became its President. The writer feels sure that those who have succeeded to the control of this corporation after the deaths of Messrs. William E. Dodge, Senior and Junior, and of Mr. D. Willis James, will not resent the statement that, in the writer's opinion, Dr. Douglas supplied the imagination necessary in all great enterprises, while they supplied the money and equally important careful business management.

The product of the smelter at Bisbee was hauled several miles to the railroad by mules. He put in the first traction engines employed in the Southwest. This method becoming too slow, he built the railroad from Bisbee to Fairbanks, the junction with the Southern Pacific. When the product of the Copper Queen became too great to handle economically at Bisbee, it was his idea to establish at Douglas the beginning of the great smelting plant which today is second to none—if not in capacity, at least in well thought-out installation and correlation of its parts; in efficiency and economy in management.

Looking further ahead than the life of the Copper Queen, it was Dr. Douglas who suggested the taking over of adjoining properties in the Bisbee camp, and the agreement to disregard the law of the apex and questions of extra-lateral right, so there has been no litigation at Bisbee from these fertile sources of trouble in most mining camps.

It was Dr. Douglas again, when fuel became expensive and irregular in delivery, who suggested the organizing of a coal company to supply their own needs and to enable them to sell coal and coke to others without paying tribute in high freights to the railroad. Again, it was his suggestion that their railroad should be extended to El Paso, and that branch lines should be built into Mexico, where, on his initiative, Phelps, Dodge & Co. had already secured important producing mines, destined to add a very considerable tonnage to their output of copper.

In 1875, the year when Douglas came to Phoenixville, he was elected a member of this Institute, and in June, 1876, he entertained at his works a visiting party of its members. Already, thus early in his career, he manifested the quality which was afterward so characteristic of him—a great willingness to communicate, as well as to receive, the results of discovery and practice. The Institute, founded to promote this open interchange of professional knowledge, appealed peculiarly to his mind, which disdained to harbor secrets. Through the five years in which he bravely fought at Phoenixville a losing fight, he remained in the Institute; but when that enterprise had failed, and he was obliged to begin again somewhere else, he resigned his membership, the annual expense of which he could not conscientiously afford. But after professional

recognition and business success had returned to him, he secured reinstatement in that relationship and thenceforth unto the end was in every way a loyal, potent and munificent supporter of the Institute. I mention this episode of his temporary retirement, because it was influential in the history of the Institute itself. For years after Dr. Douglas had become a leader in its management (he was Vice-president in 1897-8, President in 1899-1900, Director from 1905 to 1913, Honorary Member from 1906, and Vice-president of the Board of Directors from 1906 to 1911) he was a strong opponent of the increase of annual dues. Even after the acceptance by the Institute of Mr. Carnegie's gift (involving a heavy land-debt), he was not willing to meet additional expenses in that way. "I remember," he used to say, "when even ten dollars a year was too great a burden for me; and I will not vote to lay a heavier load upon young American engineers." I promised, as Secretary, to take care of all office expenses out of the annual dues as they stood; and that promise was kept. But it was not possible to make payments also upon the land-debt; and we proposed to pay that by subscription. This is, indeed, what was finally done; and, without disparaging the liberality of other subscribers, it must be said that the early success of the movement, which paid the whole debt (\$180,000 and interest) ten years before the final payment was due under the terms of the mortgage, was chiefly the work of James Douglas. It is true, that after this achievement, the Institute was unable to meet its running expenses without raising the dues to \$12, and Mr. Douglas was beaten on that point, after all. But the whole story shines with his generous and sympathetic courage.

One day, in a private talk over Institute affairs, I would have cautioned him against a too reckless giving away of his money; but he cut me short by saying, "This year, for the first time in my life, I have what I may call a large—really, Raymond, a very large—income; and I mean to get some pleasure out of it!"

At the time when Phelps, Dodge & Co. became a corporation, the members of the firm set aside \$10,000 as a gift to Dr. Douglas, in recognition of his past services. But under his earnest persuasion, they gave the money to the Institute land-fund instead, in addition to their earlier subscription of some thousands of dollars. He was himself also at that time already a large subscriber.

This does not exhaust the list of his benefactions to the Institute. After the land-fund had been completed, he gave \$100,000 to the Library; and it is reported in the newspapers that he left to the same object \$100,000 more in his will.

Dr. Douglas's contributions to our *Transactions* are shown in the following table:

## PAPERS

Title	Vol.	Page	Year
The Copper Resources of the United States.....	xix	678	1890
Biographical Notice of Thomas Sterry Hunt.....	xxi	400	1892
Summary of American Improvements and Inventions in Ore-crushing and Concentration, and in the Metallurgy of Copper, Lead, Gold, Silver, Nickel, Aluminum, Zinc, Mercury, Antimony and Tin.....	xxii	321	1893
Note on the Operation of a Light Mineral Railroad.....	xxviii	600	1898
Notes on the Stockholm Exposition and the Iron and Steel Trade of Sweden.....	xxviii	101	1898
American Transcontinental Lines.....	xxix	782	1899
The Characteristics and Conditions of the Technical Progress of the Nineteenth Century.....	xxix	648	1899
The Copper Queen Mine, Arizona.....	xxix	511	1899
Biographical Notice of William Earl Dodge.....	xxxiv	413	1903
Secrecy in the Arts.....	xxxviii	455	1907
Conservation of Natural Resources .....	xl	419	1909

## DISCUSSIONS

Summary of American Improvements and Inventions in Ore-crushing and Concentration, and in the Metal- lurgy of Copper, Lead, Gold, Silver, Nickel, Aluminum, Zinc, Mercury, Antimony and Tin.....	xxii	647	1893
Stockholm Exposition and the Iron and Steel Trade of Sweden .....	xxviii	813	1898
The Copper Queen Mine, Arizona.....	xxix	1056	1899
Corrosion of Water-jackets of Copper Blast Furnaces ...	xxxviii	879	1907
Coal-briquette Plant at Bankhead, Alberta, Canada.....	xxxix	894	1908
Conservation of Natural Resources.....	xl	878	1909

Besides these professional papers, and many of the same class read before other technical societies, Dr. Douglas published the following books:

*Canadian Independence, Annexation and Imperial Federation.*

*Old France in the New World.*

*New England and New France.*

*Journal and Reminiscences of James Douglas, M.D., Edited by his son.*

He received the degree of LL.D. from both Queen's University (of which he was Chancellor when he died) and McGill University, of which he had been a trustee for many years. In 1906, the gold medal of the Institution of Mining and Metallurgy of Great Britain, and in 1916 the John Fritz gold medal, were awarded to him. In 1906, as already mentioned, he was elected an Honorary Member of this Institute, and in 1907 the same title was conferred upon him by the Mining and Metallurgical Society of America. He was a member of the American Philosophical Society, the American Geographical Society, the Society of Arts, of London, the Iron and Steel Institute, and other learned and professional bodies.



Besides his munificence toward this Institute, Dr. Douglas was a liberal benefactor of many good causes and institutions and of innumerable individuals. A few instances only can be mentioned here. A much larger number will never be publicly known. In addition to his generous gifts during his life, he bequeathed \$100,000 to the American Museum of Natural History, \$100,000 to this Institute, \$100,000 to the General Hospital at Kingston, Canada, and \$50,000 to McGill University, besides legacies to many employees.

In short, he lived long enough to realize the purpose of his life, and to "get some pleasure out of it."

Dr. Douglas in 1860 married Miss Naomi Douglas, daughter of Captain Walter Douglas, of Quebec, who survives him, together with two sons and two daughters: Major James F. Douglas, developer of the United Verde Extension Mine, who is now serving in France; Walter Douglas, who succeeded his father as President of the Phelps-Dodge Corporation; Mrs. Edith M. Douglas, wife of Archibald Douglas, a New York lawyer of extensive mining interests; and Miss Elizabeth Douglas.

Good-by for a while, James Douglas!—unwearied worker, courageous leader, wise counsellor, glad giver, faithful lover and friend—"Douglas, Douglas, tender and true!"

### Biographical Notice of Edward Dyer Peters\*

Edward Dyer Peters, the only child of Henry Hunter Peters and Susan Barker Thaxter, was born in Dorchester, Mass., June 1, 1849. From his father he was a descendant of the Peters family of Ipswich and Andover, Mass., whose earliest ancestor settled in Essex County in 1659; while upon his mother's side he descended from several old Hingham families, as well as from the Quincys and from Governor Bradford.

Between the ages of fourteen and sixteen he was a student at the Episcopal School for boys in Cheshire, Conn. Near the school was an old tin mine, and he used to spend his Saturday afternoons exploring it.

When Edward was sixteen, his father sold his property in Southborough, and for the next three years lived in Europe. Sept. 26, 1865, Edward entered the Royal School of Mines at Freiberg, Saxony.

In the summer of 1867 he joined his family on Lake Geneva, and spent a part of the season in that vicinity, going to Chamounix and taking other trips. Extract from a letter to his father:

*Elbingeroode, Harz, Aug. 30, 1868:* I left Freiberg with my friend Lilienthal, on Wednesday, the 19th of August, reached Leipzig in the evening and spent the night in that city: the next morning we took the cars and in the afternoon reached Eisleben, in Prussia, which was to be our first stopping-place; there are immense copper-mines and smelting-works at this place, and the method of working the mines is very peculiar

The next morning we got up at five and spent the whole day in the smelting-works where I obtained a number of drawings of the furnaces they use here and a good many valuable data. Saturday morning we got up at four o'clock, and descended a mine about three miles from the town; the Director of this mine was one of the most intelligent and agreeable men that I ever met; he accompanied us in the mine and explained everything that was new to us. The system of mining at Eisleben is peculiar to this town, and is not used at any other place in the world; the copper ore comes in narrow, flat veins eighteen inches high, at the utmost, consequently the men work lying flat on their left side and using the pick with both hands: the ore is carried out of the side passages into the main ones, in very low waggons about twelve inches high, and four by two feet; the boys who pull these waggons have a board buckled to their left thigh and their left arm; they pass their bare foot through a strap in the waggon and wriggle along on their side dragging the waggon after them, with their foot, almost as fast as a man can walk above ground. In the afternoon we walked over to Hittstadt, about twelve miles, and remained there Sunday and Monday. Monday night, at ten o'clock we went out to one of the smelting-works, to see the refining of the copper; this is only done at night, so we worked there from ten in the evening till eleven the next morning, and came home all blackened up and with blistered hands, for our skin was not quite so tough as that of the workmen: at noon the same day, we took the stage to Ascherleben, and then the train to Stassfurt, where there are immense salt-mines, as well as factories for the manufacture of bromium, which latter process is, however, kept a profound secret, so we had very little hope of seeing anything but the salt mines; we were talking with our landlord about our disappointment, that evening, when he told us that the director of the Bromium works was sitting in the next room with no company but his glass of beer, so we went over to him, introduced ourselves as Americans, and actually succeeded in persuading him that it could do his business no earthly hurt if he showed us his works; at last he consented, after making us give our word of honor

\* This biography was prepared from the "Biography" published by his sister, Mrs. Edward McClure Peters, in 1918.

not to divulge any of his secrets on this side of the water, and I now have, in my notebook, very accurate notes on the whole process. The next day we accomplished a good deal; we descended two mines, visited the Bromium works, and succeeded in catching the last train for Ballinstadt, in Anhalt-Bernberg. Friday morning we walked over to 'Victor's Hütte,' twelve miles, where there are very bad smelting-works, but a magnificent dressing of the ores for smelting; as this will certainly be a very important subject in the Territories, where fuel is so scarce, we devoted that afternoon and the whole of the next day to it; I filled twenty-one pages of my notebook with sketches and measurements of the different machines in use at these works; they are all remarkably simple and cheap, but extremely practical; yesterday (Saturday) I made the longest walk that I have ever made in my life; we left 'Victor's Hütte' at seven A.M. and reached this place at ten P.M., passing through the most beautiful portions of the Harz; our walk, in a straight line, measures on the large map, thirty-one English miles, but counting in all the turnings and twistings of the paths that we took, must have been at least forty. We each carried a heavy knapsack, or rather satchel, and a shawl, and when we reached this place were pretty well knocked up, so I am not at all sorry that we are to rest to-day. So far our trip has been eminently successful, and I have learned a great deal and obtained a great many valuable drawings, etc., in a very short time."

During the latter part of his residence at Freiberg he was president of the Anglo-American club, then composed of about forty-two students, and in this capacity, he was the host at a dinner given by the club to the Crown Prince of Saxony.

In the summer of 1869 he returned home, and in the fall of the same year he left for Colorado. For two or three years he was at Denver, Central City, Fairplay, Breckenridge, and Dudley.

Extract from a Colorado paper 1870 or 1871:

MIDDLE BOULDER: SILVER BULLION SHIPMENTS. The first two shipments of silver from A. D. Breed's new reduction works at Middle Boulder were made within the past five days. The first of these embraced four hundred pounds, and the last 6,200 ounces of silver, in bars, the product of ore from the Caribou mine. The bullion has been forwarded to New York . . . . Under the skillful management of such competent millmen as Charles E. Sherman and E. D. Peters, this mill will henceforth be found all that could be desired and will take the lead in Colorado in the production of silver bullion.

From another Colorado paper of a little later date (1872):

A Good Appointment: Edward D. Peters, a young man of rare abilities and acquirements, and who has few equals, theoretically or practically, in mining and milling, has been appointed and confirmed Territorial Assayer for the Southern District of Colorado, the office being located at Fairplay, Park County. At present Mr. Peters is engaged at A. D. Breed's Reduction Works, at Middle Boulder, as assayer. We congratulate him upon his appointment, and the people of the South in obtaining the services of so thorough and correct a metallurgist.

His first contributions to the *Engineering and Mining Journal* were made at this time, while he was assayer at the first Caribou Silver Mill, in Boulder County, when he sent some brief notices, on milling results, to Dr. Raymond.

From July, 1871, to March, 1872, he was superintendent and metallurgist of the largest silver mine in the country (the Caribou)—which position he resigned, having received an appointment in February, 1872, from the Governor of the Territory, as territorial assayer for the southern part of Colorado. In the fall of the same year he built the Mt. Lincoln smelting works; he writes from Dudley, February 1st, 1873.

It is a beautiful winter's morning and I can hardly realize that I am in the centre of the Rocky Mountains almost eleven thousand feet above you. My office is in a little flat, surrounded by thick pine forests, and just at the very foot of the highest peak in this whole range from British America to Mexico. The ground commences rising from my very doorstep, and in the course of one eighth of a mile, becomes so steep,

that only the little Mexican jacks can be used to pack up provisions and supplies to our mines, which are situated almost on the summit of the highest peaks. As the snow lies some five feet in the timber on the mountain sides, even they are useless now, and are down on a rancho in the valley, some fifty miles from here, dying as fast as they can, from the epizootic. We have lost some forty in the last month, and all the horses have it so badly that we only get our mails from Denver every two or three weeks, brought up by bull-teams. The smelting-furnace, which I built last fall, and for which I imagine that I have the same sort of affection that a mother has for a pet child, is running finely, without any delays or drawbacks, and is building me up quite a reputation throughout the territory. When I came here I was simply 'Professor' or 'Captain' (every man has a title out here you know). When the furnace started up successfully I was at once dubbed 'Major' and since then have usually been addressed as 'Colonel.' I expect a Brevet Generalship shortly, as promotion is rapid in this country, but if I should ever make a failure it would not take long to degrade me to the ranks. However, I am not afraid of that. The ores are easily treated. There is a fascination in the business that I am engaged in that you will not appreciate. It is certainly a pleasant trade to take the muddy ore from the mines, to determine its exact value and pay for it, to put it through the different furnaces, and finally bring out the gold, silver, copper, and lead, and above all to have the products agree exactly with the amount that you promised beforehand. There is a touch of the old alchemist's mysteries about it which renders it always new and interesting, and one feels every day that he is not only adding to the material wealth of the world, but is also filling his own mind and pocket.

In November, 1874, he returned to the East. Mining was at that time in a very low state; the ores in Colorado were supposed to be worked out, and Peters gave up all hope of continuing in his chosen profession and entered Harvard Medical School, graduating thence in 1877, and being first in a class of one hundred and sixty; this gave him the degree of Doctor of Medicine, by which he was thereafter always known. He was the only medical graduate whose thesis was mentioned on the Commencement program of that year. He practiced medicine in Dorchester from 1877 until 1880, then returned to his original profession.

On Sept. 28, 1881, he married, in Dorchester, his cousin, Anna Quincy Cushing, the daughter of his mother's sister and of Dr. Benjamin Cushing.

For some years after this date Dr. Peters was associated with and originated some of the largest American copper and nickel smelting establishments.

In the summer of 1881 he was employed by the Orford Nickel and Copper Company.

In the summer of 1882 he was at the Ely mine, Vermont, as manager, and in November of the same year he returned to the Orford Nickel and Copper Company, being the first metallurgical manager of their works at Bergen Point, New Jersey.

In 1882 and 1883 he made contributions on the metallurgy of copper to *The Mineral Resources of the United States*, and a little later contributed a series of papers to the *Engineering and Mining Journal* which in 1887 were published in book form under the title of *American Methods of Copper Smelting*, his first and probably his most valuable and important work.

In the fall of 1883 he was in Ansonia, Conn., with Mr. Franklin Farrell, of that town, and in the spring of 1884 he accompanied Mr. Farrell to Butte, Montana, where he erected the first successful concentrating and smelting works of the Parrott Silver and Copper Company.

In November of this year, 1884, he returned home, finding himself unable to face a winter in so cold a climate; he remained in Dorchester during the greater part of 1885, being engaged in some work in East

Boston, as well as in making drawings for the refining plant of the Calumet and Hecla; these plans were, however, not used.

Early in the year of 1886, Dr. Peters, having for some time felt the need of country life, and desiring some occupation beside his profession, purchased a farm at Walpole, about fifteen miles out of Boston, where he began to raise ducks with distinct success, but which he was obliged to give up, within two or three years, on account of his many absences from home and of his wife's health.

From June, 1888, until the spring of 1890, he was connected with the Canadian Copper Company, at Sudbury, Ontario, as general manager, starting the first blast furnace in December, 1888, and the second one in September, 1889, for the production of nickel copper matte.

During the following year or two he was engaged, principally, in writing, and in 1891 he published an extended, revised, and corrected edition of his first literary work, under the new title of *Modern American Methods of Copper Smelting*.

Late in the year 1892 he received a cable requesting his presence in Tasmania, to report upon the Mt. Lyell mine, and to do some preliminary work there; this mine, of iron and copper, which with the exception of the Rio Tinto mines in Spain, was the largest mine of its kind in the world, was situated in a most inaccessible locality, and required a long and fatiguing journey to reach. It was in great part owned by the members of the Great Broken Hill Mining Company, of Australia.

Dr. Peters sailed from San Francisco in December, 1892, stopping at Honolulu, Samoa, and Auckland, and encountering a severe storm between the latter place and Sydney; this was one of the worst gales experienced in that locality in nearly twenty years, and injured a number of persons on the steamer; as the ship's surgeon had left at Auckland, Dr. Peters' medical knowledge was of great use to him, and of considerable advantage to the many passengers who had received injuries. While in Melbourne he was treated with great consideration, being made an honorary member of all the clubs of consequence, and leading quite a gay life socially.

In January, 1893, he started for Tasmania, landing at Launceston, going thence by train to Hobart, then by a small steamer to the western coast, taking a waggon from their landing place, Strahan, and finally riding the last thirty miles of the journey to the Mt. Lyell mine.

"*Mt. Lyell, Tasmania, Jan. 18, 1893.* I am thirty miles from even an attempt at civilization; there is only a trail in here suitable—or rather unsuitable—for pack-animals; the mail comes in once a week, brought by a native on a mule, and though a telegraph-line runs through the bush, within a few miles of us, yet it does us no more good than if it were a clothes-line. This is just the uncivilized, uncomfortable, backwoods kind of place that I have 'sworn off' from going to, but what won't we do for money? At any rate it is safe, there being neither wild beasts nor wild men in Tasmania.

"*Jan. 11. a. m.* To-day we start to walk over the proposed line of railway that I may decide on its suitability. A long, hard, twenty mile walk, with no trail; only the bush chopped out a little. The mail arrived unexpectedly last night: I find all the local and even the Sydney and Melbourne papers, are devoting from half to two columns to my arrival, biography, etc., which latter they must get from old American journals, as I have positively refused to speak either of myself or of my mission here, and have confined myself to speaking to the reporters, solely, about copper mines in the United States. . . .

"I may have to spend some months here, for their main object, after once telling them that it will pay, is to have me put up a small plant, and smelt, and worse still, refine, a thousand or two tons of the ore, so that they can show the people in England,

to whom they intend applying for capital, to build extensive works, that the process and method of treatment are already an established success.

"May 22, 1893. . . . I am all through at Mt. Lyell at last, and really feel badly to leave it and Schlapp the superintendent, and my Zeehan and Argenton friends, Beardsley, and Elburn and all; and my engineer-in-chief Cullen, and his three or four assistants, perhaps never to see any of them again—though the Company wants me to come out again and build and start the works, etc. Fortunately a winze which I started on a promising streak of ore, when I first came to the mine, has shown up such wonderful ore that two miners took out thirty thousand dollars in silver last week.

After some stay in London, Dr. Peters went to Paris for the winter of 1893 to 1894: he worked at the École des Mines preparing for a new edition of his work on copper smelting. On behalf of the Mt. Lyell company he visited the Rio Tinto mines in Spain in September, 1893, and later, the Mansfeld mines in Germany; as well as Freiberg in the spring of the following year.

Dr. Peters spent much of the summer of 1894 in the Western United States on behalf of the Mt. Lyell company, as well as in getting data for the rewriting of the seventh and very much enlarged edition of his book, which was issued in 1895 under the title of *Modern Copper Smelting*.

For some years thereafter, Dr. Peters was engaged in various mining enterprises, the most important of which was the opening of Las Esperanzas coal mines in Mexico.

Subsequent to his work in Mexico, Dr. Peters was in Missouri,—at the Cornwall copper mines, Ste. Genevieve,—and in Georgia. In 1901 he made a short business trip to Europe, and that winter he delivered a course of lectures at the School of Mines, Columbia University. In the spring of 1904, he was invited by President Eliot, of Harvard, to give a course of lectures on the metallurgy of copper, and in the fall of that year he was appointed Professor of Metallurgy at Harvard University. The summer of 1905 he passed in Europe, upon a pleasure trip, and in 1907 he published his second great work, *Principles of Copper Smelting*. In 1909 he was appointed Gordon McKay Professor of Metallurgy. In February, 1910, he was one of a commission to make the annual assay of the coin of the United States, at the Mint, at Philadelphia. In 1911 he published *The Practice of Copper Smelting*, which he wrote "to replace his former book entitled *Modern Copper Smelting*," although the demand for the latter kept up to such an extent that the publishers continued to print it.

In 1914 he bought a farm in Shirley, Massachusetts, and there spent the remaining summers of his life; he engaged in the raising of poultry and took great pleasure in this occupation and in motoring through the surrounding country.

In March, 1914, he delivered an address, before the Canadian Mining Institute, at Montreal, upon "The Production of Heat in Metallurgical Furnaces."

During the last year of his life he was not in his usual health; he died, very suddenly and peacefully, at his own home, upon the morning of the seventeenth of February, 1917, having been able to attend to his ordinary work up to the very last day of his life.

He was a member of the Harvard, the St. Botolph, and the Papyrus Clubs of Boston, of the Engineers Club of New York, and one of the earliest members of the American Institute of Mining Engineers (1872), of which he was at one time vice-president; he also belonged to the American

Academy of Arts and Sciences, the American Association for the Advancement of Science, of which he also became a fellow in 1916, and to the American Geographical Society. He had received the degree of Doctor of Medicine from Harvard in 1877, and that of Dr. Ing. (Hon.) from the Royal School of Mines, Freiberg, Saxony, in 1914. For the last two years of his life his professorship was in the Massachusetts Institute of Technology as well as at Harvard, owing to the merging of the mining departments of these two bodies.

Beside the man of science and the professor, there was, also, something of the musician in Dr. Peters' nature. In 1877, or perhaps earlier, he began the study of the 'cello, which, though interrupted by long and repeated absences, was always resumed with satisfaction, and was through many years a relaxation and a solace.

The straightforward, practical views of life expressed by Dr. Peters became crystallized in the minds of his family, in such phrases as "Face the issue"; and "There is no reason why you should have a thing because you want it": he had himself thoroughly in hand, though his heart was there for all those who needed it. A near and dear member of his family writes: "I think he was essentially lovable, rigidly honest, and *square* in all business dealings, and always ready to 'face the issue,' a favorite expression of his."

He was absorbingly interested in the present war, and, in spite of his German education, his feelings were, from the first, pro-Ally: America's share in this world conflict would have been inexpressibly satisfying to him.

## Man Power\*

BY J. PARKE CHANNING,† E. M., M. S., NEW YORK, N. Y.

(Colorado Meeting, September, 1918)

WE are accustomed to think that we are efficient in the United States, particularly with respect to such things as mining and manufacturing. The conduct of the war has demanded in England and in France a complete readjustment of manufacturing methods and plans, and today England is probably as efficient a country as there is in the world, not even excepting Germany. This is all the more remarkable because it has been notorious for years that England has been inefficient in her manufacturing and the country has been flooded with things "Made in Germany." Today England is almost a socialistic community and the State is doing almost everything. England is now in such a position that practically everyone in the country is engaged in industry necessary for the conduct of the war, and this has been accomplished by increasing the efficiency both of her tools and of her man power.

In the United States we certainly have been efficient so far as machines and perhaps so far as methods have been concerned, but we have not been efficient in the utilization of our man power. Before the war, our labor was undoubtedly far more efficient than that of England, but it certainly was not so highly efficient as it should have been, and the problem that confronts us today, and will all the more confront us after the war, is to make our man power efficient. England has had a taste of what you may call State Socialism, and her laboring men are not going to be content to return to the old order of things. There is one feature of the labor problem in England which has permitted her to reach this condition of state socialism with comparative rapidity; this is that practically all of her laborers are English; she has little or no foreign population. While an Englishman may be a strong union man and ready to fight his employer tooth and nail, at heart he is still a British subject, and when his country was in danger he rose to the occasion.

In the United States we have such an admixture of unassimilated foreigners that the problem is more difficult, and as yet we have not been brought to that point of stress which has arrived in England. But if

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\* Presented at a meeting of the Boston Section, Mar. 15, 1918.

† Vice Pres., Miami Copper Co.



we are to carry the war to a successful conclusion, and if we are to increase our efficiency after the war, we must introduce methods which will Americanize these foreigners and give them our own point of view. We have been called the melting pot, but it is a question whether even our melting is efficient, and whether at the top of the crucible there does not accumulate too much dross and at the bottom not enough clear alloy.

I wonder if any large number in this country have read the so-called reconstruction program of the British labor party. It will, of course, be subject to a great many modifications before it is adopted by the party, and no doubt still further and greater modifications will be made before any or all of it is accomplished. It is very largely socialistic and has for its basis four principles or pillars, as they choose to call them, of the house which they hope to erect. These four pillars are:

1. The universal enforcement of the national minimum.
2. The democratic control of industry.
3. The revolution in national finance.
4. Use of surplus wealth for the common good.

If these four demands are carried out, then surely England will be a socialistic state.

I am not prepared to say how much of this program can, or will, be carried out, but it shows the trend of thought of the laboring man in England. He has seen his wages increased so as to keep pace with the growing cost of living, he has seen the profiteer discouraged, and he is more than ever convinced that in the past he has not been getting his fair share of the product of his toil; and, I believe, at the same time he is realizing that undoubtedly in the past he has not done his proper share in increasing the wealth of the country. Nor can he be blamed for this, because, seeing large fortunes grow up before his eyes, while he gets but a small proportion of it, the incentive to increased efficiency has not been great. He realizes that when the war is over, unless the greatest care is used in the reorganization of the regular industries, there will be an immense amount of unemployment; that this, if unchecked or uncared for, will result in an over supply of labor, and, if the old standard is maintained, a corresponding diminution in wages. This he feels should not be; hence his insistence of the first principle of a minimum wage. And the minimum wage that he asks for is certainly not a high one, being 30s. or, we will say, \$7.50 a week.

In demanding democratic control of industry he has observed such good results attained in war work that he sees no reason why this control of industry should not be just as efficient under after-war conditions.

In the third pillar, the revolution in national finance, he demands that taxation shall be so adjusted that it will yield the necessary revenue to the Government without encroaching upon the prescribed national

minimum standard of life of any family whatsoever; without hampering production or discouraging any useful personal effort, and with the nearest approximation to equality of sacrifice. Apparently he is not a protectionist and repudiates all proposals for a protective tariff; however, this may be disguised. In this point they agree with Mr. Courtenay de Kalb, a prominent mining engineer, who, in the December, 1917, number of the *Atlantic Monthly*, has a most excellent article on the Formula for Peace, in which he states that, if after the war we can have an industrial world in which there are no protective tariffs and no subsidies, in which every nation is engaged in producing those articles for which it is best adapted, then there will be less incentive for war.

The fourth pillar of the English laborite is that the surplus wealth shall be used for the common good. They say that we have allowed the riches of our mines, the rental value of the land superior to the margin of cultivation, the extra profits of the fortunate capitalists, and even the material outcome of scientific discoveries, to be absorbed by individual profiteers, and he demands that in the future a large proportion of this surplus shall be applied to the common good.

You must realize that the English labor party is not like the Bolsheviks of Russia. It is not carried away with the beliefs of Lenine and Trotzky, that the proletariat are the men to manage the country. The English laborer frankly realizes the importance of brains and education, and admits that the highest success of the country cannot be obtained without the aid of those who plan, and manage, and invent, nor would he object to allowing these men to get their fair share of the profits. Evidently the class against which his program is aimed is comprised of those more or less sharp and shrewd men who, without anything more than commercial ability, of themselves reap the advantages of the brains and muscles of others.

It is not for me, nor am I a sufficient student of economics, to pass upon this program. It has certain merits, and I am calling it to your attention only that you may see that just this same thing is liable to come up in the United States. And the trouble will be that the pendulum will very likely swing too far if the employer class in the United States does not give more attention to the laborer and see that his condition is improved. You, as engineers, are in the position to act as the instruments for carrying out this necessary work. Whether it be in a mine or a manufacturing plant, I believe I can say that today a large proportion of the managers and executives are engineers, and the proportion is constantly increasing.

The question to be clearly faced is, are we properly trained to bring about this improvement in our social condition, to improve the living conditions of our laborers and at the same time to improve their efficiency? I fear that a great many of us are not. We may be good technical men

but we are not sociologists nor psychologists. We understand production of kilowatt-hours from coal or from water power, we understand the machine by which it is utilized, but we do not understand the machine which produces our man power.

I recently attended a conference at Columbia University, at which the question of giving the engineering students a course in human engineering was discussed, and I came away with the idea that the authorities were beginning to realize that this was of paramount importance and that this training must be given the engineering student before he can be turned out as a man capable of eventually holding a high executive position. Many of the students have the ambition to hold high positions, but at the same time, in the most naïve manner, announce that they have no desire to have anything to do with the working men themselves. In my opinion, there never was a time when it was so necessary to impress upon engineers and engineering students the importance of this human side of engineering.

I wonder how many of you can tell me what trade unionism is—you who have had to deal with unions and have had strikes? The fact is that none of you can tell what trade unionism is because trade unionism is not an entity but a term of broad generalization covering a great many distinct aspects of the labor problem. Some of you who, perhaps, are railroad superintendents, whose knowledge of trade unionism is based upon your contact with the Brotherhood of Locomotive Engineers, would give one definition; others, who have been managing a mine in the Rocky Mountains, whose contact has been with the Western Federation of Miners, would give another definition. Specifically, each of you would be right from his own point of view, but neither of the definitions would cover trade unionism as a whole. A few weeks ago I might have been rash enough to attempt a definition, but in the meantime I have read Professor Hoxie's work on "Trade Unionism in the United States," and my ideas on the subject have been much clarified. I would advise every one who has anything to do with trade unionism to get this work and not merely read it but study it as you would study a book on electric motors to find the difference between an induction motor and a synchronous motor, between one that was simply wound and one that was compound wound.

You will find that trade unionism can be classified under two broad general heads, one based on structural varieties, and one on functional varieties. As Professor Hoxie points out, there are four divisions under each head and any one of the structural varieties may function in any one of four different ways. You will learn that while, to you, decreased output on the part of the laborer seems inexcusable, yet, for him, it has an intense and immediate value. You will find the reason why he insists that the good and the poor workman shall each turn out

the same amount of product every day, and you will find that he has most excellent reasons for this, reasons that probably never entered your head. You will learn why the locomotive engineers of the United States can have one strong central national union, and why this is impossible with the men who dig ditches. You will discover why the Knights of Labor movement failed and why the American Federation of Labor has succeeded. There is one basic and most important factor which you must realize, namely, that, talk as you may, the interest of the laborer and the interest of the employer are diametrically opposed, just as the interest of the buyer and the interest of the seller are opposed, that from the very nature of things they can never be identical, and that the best that can ever be reached is a compromise. And who is better qualified to bring about this compromise than the well trained engineering manager who, with his broad knowledge and experience with both capitalist and laborer, is enabled to act as an arbiter or a judge and arrive at a decision at least fairly equitable.

It is the engineers of this country who are in a position to solve the labor problem, or at least to produce a solution as nearly ideal as possible. It is you who are to convince the employer that, in the long run, he is going to be better off by increasing the wages of his men, reducing their hours of work, and improving their living conditions. It is you who must convince the laborer that it is to his interest to work as efficiently as he can and to produce as large an output as is possible. You will have to do this by education. It is difficult to convince a laborer that by increasing his efficiency and his output he helps himself, because he only looks to immediate results. But do not be carried away with the idea that because the laboring man upholds an economic fallacy that you cannot convince him of his error.

About 10 years ago I started to develop a low-grade copper mine in Arizona. As mine superintendent I had Mr. N. Oliver Lawton, a member of this Institute, whose experience at Lake Superior has made him familiar with what is known as the one-man air drill. This is a light machine weighing about 125 lb., which can be readily set up and operated by one man. We started to use these in Arizona where, before, nothing but the larger and heavier machine, requiring two men, was in use. There was an immediate opposition from the men and we were accused of trying to throw half the normal number of miners out of work. Whenever I went through the mine I took the opportunity to tell the men that this orebody, up to that time, had not been considered ore, that it was rock, and that nobody thought it was worth exploiting; that, far from throwing one man out of work, we were giving two men jobs, that if two men had to work on a drill the cost of mining would be so high that the material would not be ore, but that if we gave each man a drill and put him to work in a separate drift, then the rock would become

ore, that these men would have employment and that a new industry would be started. About three months of this propaganda convinced the men of the truth of our claim, and in a short time it would have been impossible to get the men to go back to the old two-man drill because each man now felt that he stood on his own feet and got credit for the whole distance he drifted. This is only one example, but it indicates what can be done by education. The old-time manager or old-time superintendent would simply have said, take the job or leave it; but this is not the attitude for the modern engineer.

The assertion is made in Washington that it is difficult to get executives for war work, particularly executives who understand the handling of man power. I am told that some of the new plants for war industries have been most carefully laid out, taking into consideration the routes by which material is to arrive at the plant, its progress through the works, and its method of removal, the supply of water, coal, and other material, but in a great many cases no thought has been given to the handling of the men, to their housing, or, if they are to be brought from an adjacent town, of the method of transporting them to and from the plant. These have been left to a hit or miss adjustment after the plant was up.

For several years the industrial department of the Y. M. C. A. has had a secretary who has devoted himself almost entirely to impressing upon the engineering schools the necessity for having a course in human engineering, and in 1916, under their auspices, the first convention to discuss the human side of engineering was held in Ohio. They lay great stress upon the advantages that would accrue to engineering students if they had an insight into the mental operations of the laboring man, and this has been fostered by getting the engineering students to volunteer one or more hours of the week for instruction to laborers employed in adjacent plants. This instruction is either in the English language, in citizenship, or in athletics. A man who has volunteered for this work for a year or more, on going out into active life is a much more capable foreman than one who graduates from an engineering school and meets his first laborer somewhere on his new job.

Lately the National Americanization Committee of New York, of which Mr. Frank Trumbull, of the Chesapeake & Ohio Railroad, is Chairman, has been conducting similar propaganda, sending to the various educational institutions of the country a proposed basis of a course on industrial engineering. The Committee realized the importance of this in preparing engineering graduates for the problem of properly utilizing the man power of the country. This proposed course goes into the scope of industrial engineering, describes the problem and the field; it takes up the question of the engineering insight of the work in reference to plant building, its location, and the fundamental considerations in its construction; it takes up the management and division of the

work, the analysis of the costs, and the machinery, and the materials, and the efficiency methods. It goes into the question of employment, management, and the methods for hiring, promoting, and transferring men. It also takes up industrial welfare with the various incentives to the workman and the provisions for his health and recreation, and for the vocational training of either himself or his children. It also gives instruction in that branch which is so often neglected, and that is, conditions outside of the plant, the housing of the men, the planning of the town, and the health and recreation and education of their families. It takes up the problem of Americanization and what shall be done to make our melting pot efficient, without dross, and finally it gives him instruction as to what has been done and what should be done in legislation.<sup>1</sup>

You engineers who are college graduates should use your influence to see that courses in human engineering are introduced in your Alma Maters, if they are not already there.

Do you mining engineers realize that your training and your experience, touching as they do on all branches of engineering, fit you better for broad and important work than those in almost any other profession? How many of you have been under the necessity of developing a large mining property in some out of the way place, when everything came before you and nothing could be left to chance, where you had to see that your own town was built and provided with water works and sewers, lighting plant, and schools? You had to develop a property in a place where nothing existed and you did not have a well organized community to fall back upon with all these adjuncts provided. Only recently one of my former superintendents came into the office to tell me that he had given up a \$12,000 a year position to take one with the Government for \$3600. He did not hunt this Government job, it came after him. They asked him to go on to Washington and take a job in the Ordnance Department. They said that they had found that a mining engineer has had such varied experience, and has driven so little in ruts, that at a minute's notice he can jump from 6-in. projectiles to bailed hay.

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<sup>1</sup> See *Bulletin* No. 132, December, 1917, p. xlii.

### Use of Cripples in Industry

BY JAMES P. MUNROE,\* S. B. LITT. D., WASHINGTON, D. C.

(New York Meeting, February, 1919)

APPALLING as has been the loss of life in the last 51 months, there is one slight compensation: no longer will there be in the world a cripple, in the old meaning of the term. Men handicapped by wounds or disease, there will be, unfortunately, and in numbers beyond what the world has known since the wars of Napoleon; but neither they nor the industries from which they were called off to war will be "crippled" in the sense in which both would have been had mankind not learned the lesson of conservation and come to understand that the most important field for such conservation is not in the forests and the mines but among men and women.

From the beginning of the Great War, France, Great Britain, Belgium and most of the other Allies have studied the problem of restoring the soldiers and sailors injured through war to physical and economic efficiency; and from their experiences, especially from that of Canada, the United States has learned much. Consequently, our task of preparing for the return of our disabled men has been easier and, in some ways, more comprehensive than theirs. Complex as are the details of the machinery which the United States has set in motion to take care of the men injured by wounds or disease, the plan itself is simple. Taught by European experience, the Surgeon-General of the Army and the Bureau of Medicine and Surgery of the Navy have provided, on both sides of the Atlantic, every known surgical and medical facility for restoring the injured or diseased man to a physical condition as nearly normal as possible. While in the hospital in France or England, on the transport coming to America, and in the hospital here, the disabled man is incited in every way to believe in his future efficiency, to want to be a normal worker, to desire to retake his place in that society of workers from which he went, temporarily, to do the greater work of preserving civilization. Furthermore, since purposeful occupation is now regarded as an essential form of treatment with most men in the hospital, especially in the convalescing stage, many of these men will have been actually started on the road to earning before they are discharged from the army surgeon's care.

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\* Vice-chairman, Federal Board for Vocational Education.

As soon as it is decided that a patient is ready for discharge from the hospital—and, now that hostilities have ceased, from the Army and Navy itself—his case is certified to two bodies: the War Risk Insurance Bureau, which is to determine the amount, if any, of his compensation under the War Risk Act, and the Federal Board for Vocational Education, which stands ready to help him to get back into employment and, if he needs it, to secure a preliminary training that will enable him to make the most of himself, under the conditions of his handicap, in that employment.

The Federal Board has no authority over the man thus placed under its care; it is for him to decide whether or not he wishes to avail himself of the help that the Federal Government thus offers. But if he chooses to use the facilities tendered by the Board, there is almost no limit, within reason, to what that organization may undertake for him. Its simplest task is, of course, to assist him in getting back into his old employment; but if he has ambition to get something better or if it is apparent that, by training, he can be more efficient in what he did before, the Board has authority to give him, at Government expense, as much education as, in its opinion, it is worth while for him to have. Every endeavor will be made to train the disabled man so that not only may his handicap be overcome, but that he may be carried, through an education perhaps denied to him before going to war, to a plane of efficiency which, without this opportunity, he could not have reached. Experience in other countries has shown that, in many instances, the disabled man is, after training and despite his handicap, a much more effective man than he was before the war.

While the disabled soldier or sailor is under no compulsion to take training, there are certain incentives, besides that of ambition, which the Government puts before him. If he desires to be trained and the Federal Board believes that he will profit by it, he is so certified to the War Risk Insurance Bureau, which at once classes him as entitled, during training, to the compensation provided for cases of temporary total disability, and, during the period of training, makes specified allotments to his dependents, should he have them. If he does not pursue the course of training with due diligence, these extra compensations, on representation of the Federal Board, may be withdrawn.

Training will be carried on in public and private schools and colleges and in industrial plants under contracts made between them and the Federal Board. The period of training will be determined to meet the needs of each case, but in every instance the disabled man is to be regarded as a special problem and the instructional work given him will be fitted to his needs. It will be attempted, as far as possible, to obtain for him a position in advance of his being ready for it, so that his training may be focused upon a specific goal. Should it prove, after employment, that his choice was unwise, the Board has authority to give him further



training along that, or some new line. Moreover, after placement, whether with or without training, the Board will keep closely in touch with the man until it feels certain that he is firmly established in his industrial, commercial, or professional life.

To carry out the duty placed on it by Congress, the Federal Board has established, or is establishing, headquarters, in Washington and thirteen of the other leading cities of the country. As far as possible, the disabled man will be placed and trained in his own State and locality. Every effort will be made to put him into occupations that are growing, and so to train him that, when hard times come and the fervor of patriotism has passed, he will be retained, not because he is a former soldier or sailor but because he is a workman necessary to the work. Care will be taken, moreover, that he is not exploited and that he is not used as an instrument to disturb the labor situation. The complicated problems that might arise, in many States, in connection with employer's liability laws will not come up, since the number of disabled men is happily much less than it seemed probable that the United States would have.

The comparative smallness of the problem in the case of men injured in the pursuit of war serves but to emphasize the greatness of the number of men and women injured every year in the pursuit of the activities of peace. By the hundreds of thousands they meet with accident and injury in every degree and form. Heretofore, most of these injured persons, so far as their economic usefulness is concerned, have been thrown on the scrap-heap of society, with anguish to themselves and their relatives, with incalculable loss to the community. The war has taught us that this waste is needless and wrong; and if a bill now before the Congress becomes law, the Federal Board will be charged with providing, in coöperation with the several States, facilities for training and retraining these victims of industry along the same general lines as those followed with the victims of war. The task will be far greater than in the case of the disabled soldiers and sailors; it will not be, as with them, a comparatively temporary responsibility. It will go on forever, as long as there are machines, carelessness, and the inevitable lapses in human minds and senses, and the problem will have many complications that do not arise with those disabled in war. But the effects of rehabilitation in the field of industry will be as much broader in their final results as the scope of the permanent and normal arts of peace is greater than that of the temporary and abnormal art of war.

## DISCUSSION

JAMES P. MUNROE.—The present situation is vastly different from the situation at the time the paper was written. We now know pretty well what our problem with the disabled soldier is: 50,000 disabled men have come back and the Surgeon General tells us that probably 35,000

more are coming over in the months of March and April. So the total number of disabled men will be 85,000, which of course includes a great number of slightly disabled. If we followed the proportion that obtains in other countries, we would have about 10 per cent. of those people to be retrained under the Vocational Law. As a matter of fact, the general intelligence of our men is so much higher and the provision the government has made for the training of these disabled men is so much more liberal than with the other governments that we feel very certain indeed that instead of having 8500 men to be retrained there will probably be twelve and possibly fifteen thousand.

### Mental Tests in Industry\*

PRESENTED BY ROBERT M. YERKES, WASHINGTON, D. C.

(New York Meeting, February, 1919)

THE following is a brief account of the methods of measuring intelligence especially prepared for use in the U. S. Army, of typical results, and of some of their immediately practical applications. It has been prepared for the assistance of army examiners and for the information of all who are interested in the relations of scientific placement to military efficiency.

#### PURPOSE OF THE INTELLIGENCE TESTS

Under the direction of the Division of Psychology, Medical Department, and in accordance with provisions of War Department General Order No. 74, mental tests are given recruits during the 2-wk. detention period. These tests provide an immediate and reasonably dependable classification of the men according to general intelligence. Their specific purposes are to aid:

1. In the discovery of men whose superior intelligence suggests their consideration for advancement;
2. In the prompt selection and assignment to development battalions of men who are so inferior mentally that they are suited only for selected assignments;
3. In forming organizations of uniform mental strength where such uniformity is desired;
4. In forming organizations of superior mental strength where such superiority is demanded by the nature of the work to be performed;
5. In selecting suitable men for various army duties or for special training in colleges or technical schools;
6. In the early formation of training groups within regiment or battery in order that each man may receive instruction and drill according to his ability to profit thereby;
7. In the early recognition of the mentally slow as contrasted with the stubborn or disobedient;
8. In the discovery of men whose low-grade intelligence renders them either a burden or a menace to the service.

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\* Published with the approval of the Surgeon General of the Army, from the Division of Psychology, Medical Department, Major Robert M. Yerkes, Chief. This article, in slightly different form, was printed in November, 1918, by the National Research Council.

## NATURE OF TESTS

The tests were prepared by a committee of the American Psychological Association and of the National Research Council. Before being ordered into general use they were thoroughly tried out in four National Army Cantonments. From time to time they have been revised to increase their practical usefulness. Up to January 1, 1919, approximately one million seven hundred thousand men had been tested. Three systems of test are now in use:

1. *Alpha*.—This is a group test for men who read and write English. It requires only 50 min., and can be given to groups as large as 500. The test material is so arranged that each of its 212 questions may be answered without writing, merely by underlining, crossing out, or checking. The papers are later scored by means of stencils, so that nothing is left to the personal judgment of those who do the scoring. The mental rating which results is therefore wholly objective.

2. *Beta*.—This is a group test for foreigners and illiterates. It may be given to groups of from 75 to 300 and requires approximately 50 min. Success in Beta does not depend on knowledge of English, as the instructions are given by pantomime and demonstration. Like Alpha, it measures general intelligence, but does so through the use of concrete or picture material instead of by the use of printed language. It also is scored by stencils, and yields an objective rating.

3. *Individual Tests*.—Three forms of individual test are used: The Yerkes-Bridges Point Scale, the Stanford-Binet Scale, and the Performance Scale. An individual test requires from 15 to 50 min. The instructions for the Performance Scale are given by means of gestures and demonstrations, and a high score may be earned in it by an intelligent recruit who does not know a word of English.

All enlisted men are given either Alpha or Beta according to their degree of literacy. Those who fail in Alpha are given Beta, and those who fail to pass in Beta are given an individual test. As a result of the tests, each man is rated as A, B, C+, C, C-, D, D-, or E. The letter ratings are reported to the Interviewing Section of the Personnel Office, and are there copied on the qualification cards (in the square marked Intelligence). The psychological report, after the grades have been copied on the qualification cards, is forwarded from the Interviewing Section to the Mustering Section of the Personnel Office, where each soldier's letter rating is copied on the second page of his service record. A copy of the psychological report is also sent by the psychological examiner to the company commander, who uses it in the organization of his company. In some camps, the entering of intelligence grades on service records has been left to company commanders, but accuracy and uniformity is secured by having these grades entered in the Muster-

ing Section of the Personnel Office when the service records are being started.

The psychological staff in a camp is ordinarily able to test 2000 men per day and to report the ratings to the Personnel Office within 24 hr. Personnel adjutants coöperate in arranging the schedule of psychological examinations so as to secure from them maximum value.

#### EXPLANATION OF LETTER RATINGS

The rating a man earns furnishes a fairly reliable index of his ability to learn, to think quickly and accurately, to analyze a situation, to maintain a state of mental alertness, and to comprehend and follow instructions. The score is little influenced by schooling; some of the highest records have been made by men who had not completed the eighth grade. The meaning of the letter ratings is as follows:

*A. Very Superior Intelligence.*—This grade is ordinarily earned by only 4 or 5 per cent. of a draft quota. The A group is composed of men of marked intellectuality. A men are of high-officer type when they are also endowed with leadership and other necessary qualities.

*B. Superior Intelligence.*—B intelligence is superior but less exceptional than that represented by A. The rating B is obtained by eight to ten soldiers out of a hundred. The group contains many men of the commissioned-officer type and a large amount of non-commissioned officer material.

*C+. High Average Intelligence.*—This group includes 15 to 18 per cent. of all soldiers. It contains a large amount of non-commissioned officer material with occasionally a man whose leadership and power to command fit him for commissioned rank.

*C. Average Intelligence.*—Includes about 25 per cent. of soldiers. Excellent private type with a certain amount of fair non-commissioned officer material.

*C-. Low Average Intelligence.*—Includes about 20 per cent. While below average in intelligence, C- men are usually good privates and satisfactory in work of routine nature.

*D. Inferior Intelligence.*—Includes about 15 per cent. of soldiers. D men are likely to be fair soldiers, but they are usually slow in learning and rarely go above the rank of private. They are short on initiative and so require more than the usual amount of supervision. Many of them are illiterate or foreign.

*D- and E. Very Inferior Intelligence.*—This group is divided into two classes: D- men, who are very inferior in intelligence but are considered fit for regular service, and E men, those whose mental inferiority justifies their recommendation for development battalion, special-service organization, rejection, or discharge. The majority of D- and E men are below 10 years in mental age.

The immense contrast between A and D— intelligence is shown by the fact that men of A intelligence have the ability to make a superior record in college or university, while D— men are of such inferior mentality that they are rarely able to go beyond the third or fourth grade of the elementary school, however long they attend. Many of them are of the moron grade of feeble-mindedness. B intelligence is capable of making an average record in college, C+ intelligence cannot do so well, while mentality of the C grade is rarely capable of finishing a high-school course.

#### DIRECTIONS FOR THE USE OF INTELLIGENCE RATINGS

In using the intelligence ratings the following points should be borne in mind:

1. The mental tests are not intended to replace other methods of judging a man's value to the service. It would be a mistake to assume that they tell us infallibly what kind of soldier a man will make. They merely help to do this by measuring one important element in a soldier's equipment, namely, intelligence. They do not measure loyalty, bravery, power to command, or the emotional traits that make a man "carry on." However, in the long run these qualities are far more likely to be found in men of superior intelligence than in men who are intellectually inferior. Intelligence is probably the most important single factor in soldier efficiency, apart from physical fitness.

2. Commissioned-officer material is found chiefly in the A and B groups, although, of course, not all high-score men have the other qualifications necessary for officers. Men below C+ should not be accepted as students in Officers' Training Schools unless they possess exceptional power of leadership and ability to command.

3. Since more than one-fourth of the enlisted men rate as high as C+, there is rarely justification for going below this grade in choosing non-commissioned officers. This is especially the case in view of the likelihood of promotion from non-commissioned to commissioned grade. Even apart from considerations of promotion, it is desirable to avoid the appointment of mentally inferior men (below C) as non-commissioned officers. Several careful studies have shown that C— and D sergeants and corporals are extremely likely to be found unsatisfactory. The fact that a few make good does not justify the risk taken in their appointment.

4. Men below C+ are rarely equal to complicated paper work.

5. In selecting men for tasks of special responsibility, the preference should be given to those of highest intelligence rating who also have the other necessary qualifications. If they make good, they should be kept on the work or promoted; if they fail, they should be

replaced by men next on the list. To aid in selecting men for occupational assignment, data have been gathered on the range of intelligence scores found in various occupations. This material has been placed in the hands of the Personnel Officers for use in making assignments. It is suggested that those men who have an intelligence rating above the average in an occupation should be the first to be assigned to meet requirements in that occupation. After that, men with lower ratings should be considered.

6. In making assignments from the depot brigade to permanent organizations, it is important to give each unit its proportion of superior, average, and inferior men. If this matter is left to chance, there will inevitably be weak links in the army chain. Exception to this rule should be made in favor of certain arms of the service which require more than the ordinary number of mentally superior men, *e. g.*, signal corps, machine gun, field artillery, and engineers. These organizations ordinarily have about twice the usual proportion of A and B men and very much less than the usual proportion of D and D- men.

The first two columns in the accompanying table illustrate the distribution of intelligence ratings typical of infantry regiments and also the extreme differences in the mental strength of organizations which are built up without regard to these ratings. The last column to the right shows a balanced distribution of intellectual strength which might have been made to each of these two regiments.

*Typical Distribution of Intelligence Ratings in Infantry Regiments*

Intelligence Rating	Interpretation	Two Actual Distributions		Balanced Distribution, Per Cent.
		1st Regt., Per Cent.	2d Regt., Per Cent.	
A	Very superior.....	1.0	6.0	3.5
B	Superior.....	3.0	12.0	7.5
C+	High average.....	7.0	20.0	13.5
C	Average.....	15.0	28.0	21.5
C-	Low average.....	25.0	19.0	22.0
D	Inferior.....	31.0	13.0	22.0
D-	Very inferior.....	18.0	2.0	10.0

Unless intelligence is wisely distributed certain companies which train much more slowly than others will delay the program of the regiment.

7. D and D- men are rarely suited for tasks which require special skill, resourcefulness or sustained alertness. It is also unsafe to expect D, D-, or E men to read or understand written directions.

8. Only high-score men should be selected for tasks which require quick learning or rapid adjustments.

9. It should not be supposed that men who receive the same mental rating are necessarily of equal military worth. A man's value to the service should not be judged by his intelligence alone.

10. The intelligence rating is one of the most important aids in the rapid sorting of the masses of men in the depot brigade. In no previous war has so much depended on the prompt and complete utilization of the mental ability of the individual soldier. It is important, therefore, that the psychological ratings be regularly used as an aid in the selection, assignment, and classification of men.

### EVIDENCE THAT THE TESTS MEASURE MILITARY VALUE

It has been thoroughly demonstrated that the intelligence ratings are useful in indicating a man's probable value to the service. The data on this matter presented in the accompanying diagrams are typical.

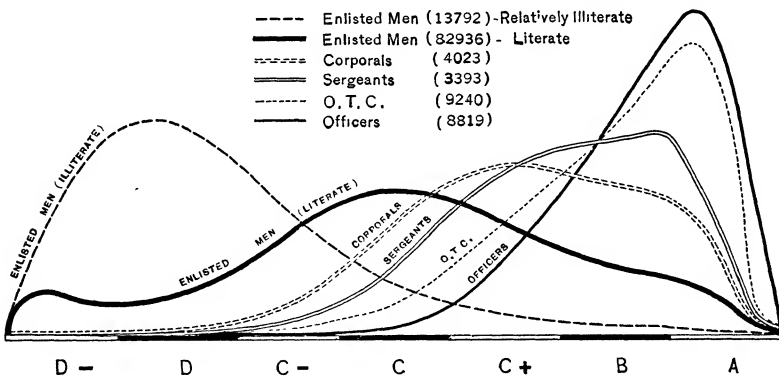


FIG. 1.—DISTRIBUTION OF INTELLIGENCE RATINGS IN TYPICAL ARMY GROUPS, SHOWING VALUE OF TESTS IN IDENTIFICATION OF OFFICER MATERIAL. ILLITERATE GROUP GIVEN BETA; OTHER GROUPS, ALPHA.

The psychological ratings have proved valuable not so much because they make a better classification than would come about in the course of time through natural selection, but chiefly because they greatly abbreviate this process by indicating immediately the groups in which suitable officer material will be found, and at the same time those men whose mental inferiority warrants their elimination from regular units in order to prevent retardation of training. Speed counts in a war that costs \$50,000,000 per day. Fig. 1 shows the distribution of intelligence ratings in typical army groups.

Fig. 2 shows the results for three Officers' Training Schools, having



a total enrollment of 1375. Note the rapid increase in elimination in grades below B. Of those above C+, 8.65 per cent. were eliminated; of those below C+, 58.27 per cent.

Fig. 3 shows the results for four Non-commissioned Officers' Training Schools, having a total enrollment of 1458. Note the rapid increase in elimination in the grades below C. Of those above C, 18.49 per cent. were eliminated; of those below C, 62.41 per cent.

In Fig. 4, note the striking intellectual contrast between those who have been selected as officer material and the men who have been designated as unteachable or of low military value.

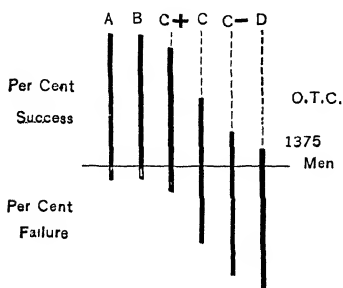


FIG. 2.—SUCCESS AND FAILURE IN OFFICERS' TRAINING SCHOOLS.

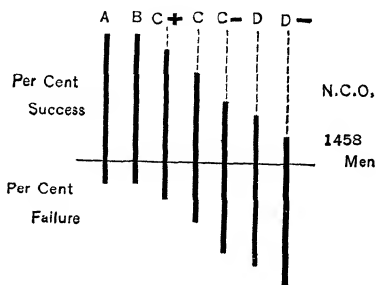


FIG. 3.—SUCCESS AND FAILURE IN NON-COMMISSIONED OFFICERS' TRAINING SCHOOLS.

The men of Fig. 5 numbered 374. They were classified in five groups by their officers on the basis of general value to the service. The men were selected from 12 different companies, on the basis of officers' knowledge of them. Approximately 30 men in each company were ranked in serial order from best to poorest by a superior officer. This rank order for each company was then correlated with the rank order furnished by the tests. In 7 of the 12 companies the correlations were between 0.64 and 0.75. The average correlation for the 12 companies was 0.536. These correlations are high, considering the large number of factors which may enter to determine a man's value to the service.

#### EXPLANATION OF FIG. 6

Commanding officers of 10 different organizations, representing various arms in a camp, were asked to designate: The most efficient men in the organization; men of average value; men so inferior that they were barely able to perform their duties. The officers of these organizations had been with their men from 6 to 12 mo. and knew them exceptionally well. The total number of men rated was 965, about equally divided among best, average, and poorest. After the officers' ratings

had been made, the men were given the psychological test. Comparison of test results with officers' ratings showed:

1. That the average score of the best group was approximately twice as high as the average score of the poorest group.

2. That of men testing below C-, 70 per cent. were classed as poorest and only 4.4 per cent. as best.

3. That of men testing above C+, 15 per cent. were classed as poorest and 55.5 per cent. as best.

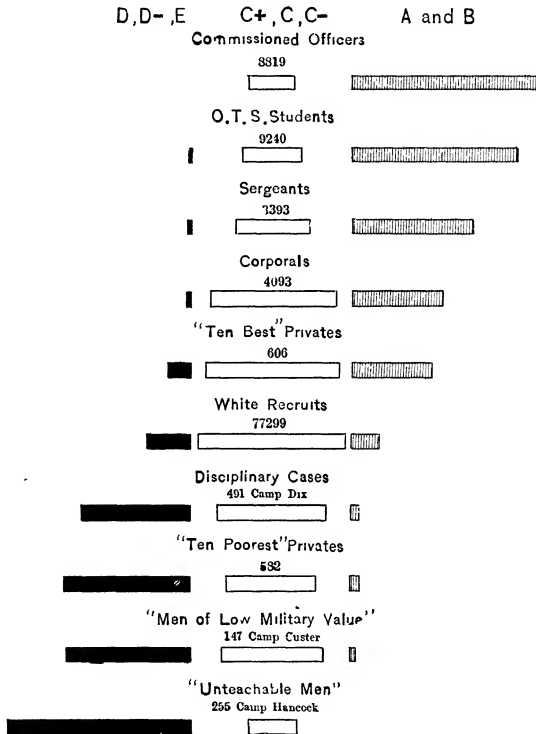


FIG. 4.—PROPORTION OF LOW, AVERAGE AND HIGH-GRADE MEN IN TYPICAL GROUPS.

4. That the man who tests above C+ is about 14 times as likely to be classed best as the man who tests below C-.

5. That the per cent. classed as best in the various groups increased steadily from 0 per cent. in D- to 57.7 per cent. in A, while the per cent. classed as poorest decreased steadily from 80 per cent. in D- to 11.5 per cent. in A. Considering that low military value may be due to many things besides inferior intelligence, the above findings are very significant.

In an infantry regiment of another camp were 765 men (regulars) who had been with their officers for several months. The company

commanders were asked to rate these men as 1, 2, 3, 4, or 5 according to practical soldier value, 1 being highest and 5 lowest. The men were then tested, with the following results:

1. Of 76 men who earned the grade A or B, none was rated 5 and only 9 were rated 3 or 4.

2. Of 238 D and D- men, only one received the rating 1, and only 7 received a rating of 2.

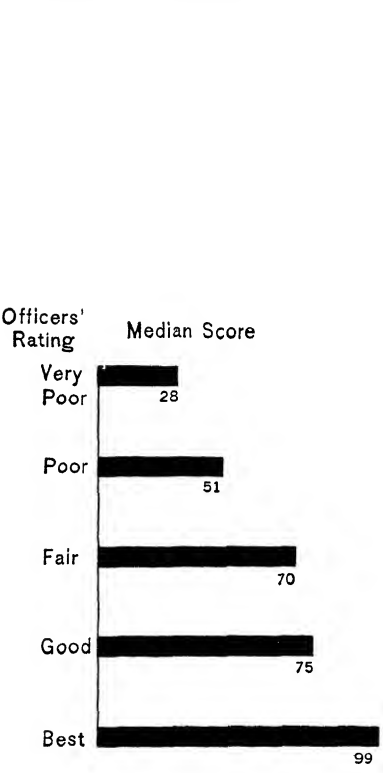


FIG. 5.

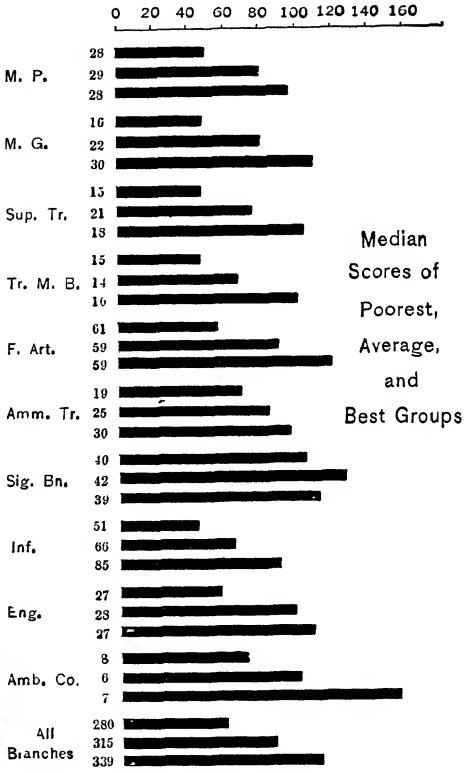


FIG. 6.

FIG. 5.—MEDIAN INTELLIGENCE SCORES (BY POINTS) OF GROUPS DESIGNATED AS BEST, GOOD, FAIR, POOR, AND VERY POOR IN MILITARY VALUE.

FIG. 6.—MEDIAN INTELLIGENCE SCORES (BY POINTS) OF POOREST, AVERAGE, AND BEST MEN IN VARIOUS ARMS OF SERVICE.

3. Psychological ratings and ratings of company commanders were identical in 49.5 per cent. of all cases. There was agreement within one step in 88.4 per cent. of cases, and disagreement of more than two steps in only 0.7 per cent. of cases.

Sixty company commanders were asked to name ten best and ten poorest privates. Of the poorest, 57.5 per cent. graded D- or D, and less than 3 per cent. A or B, see Fig. 7. The data show that a man above C+ is from 8 to 12 times as likely to be best as to be poorest; and that a

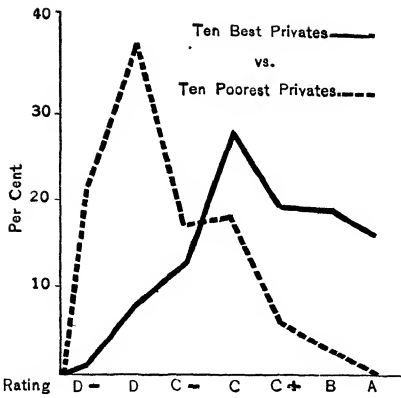


FIG. 7.

FIG. 7.—INTELLIGENCE GRADES OF BEST AND POOREST PRIVATES (BEST, 606; POOREST, 582; TOTAL, 1188).

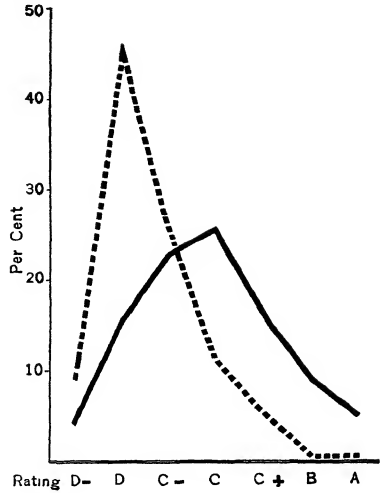


FIG. 8.

FIG. 8.—MEN OF LOW MILITARY VALUE (147) COMPARED WITH A COMPLETE DRAFT QUOTA, 12,341.

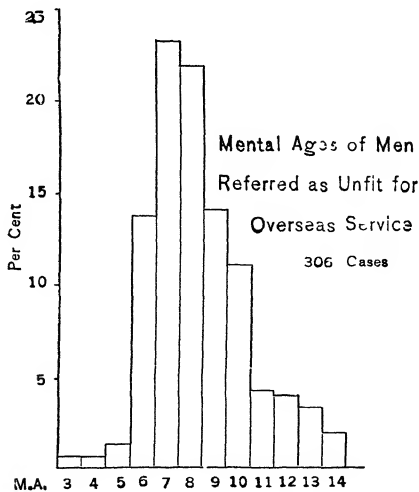


FIG. 9.—MENTAL AGES OF 306 MEN REFERRED AS MENTALLY UNFIT FOR OVERSEAS SERVICE.

MENTAL TESTS IN INDUSTRY

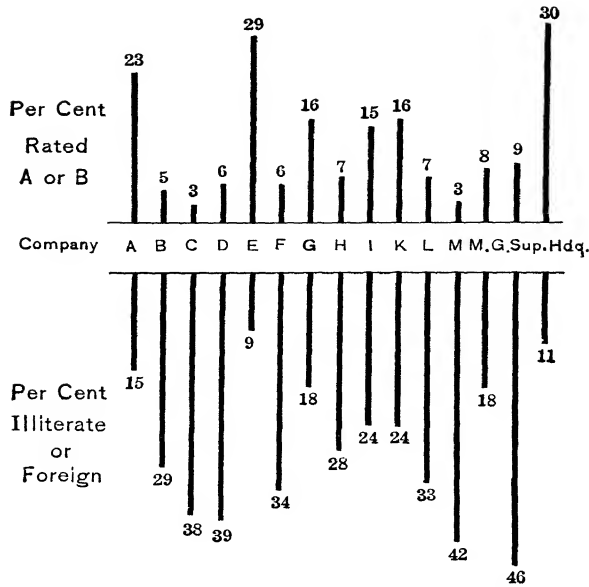


FIG. 10.—INEQUALITY OF COMPANIES IN AN INFANTRY REGIMENT.

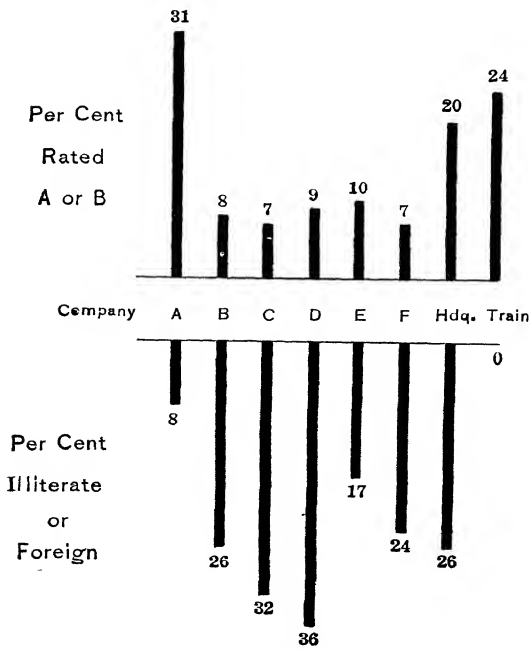


FIG. 11.—INEQUALITY OF COMPANIES IN AN ENGINEERING REGIMENT

man below C— is from 6 to 10 times as likely to be poorest as to be best. Intelligence seems to be the most important single factor in determining a soldier's value to the service.

In another camp 221 inapt men of a negro pioneer infantry regiment were referred by their commanding officers for special psychological examination. Of the 221, nearly half (109) were found to have a mental age of 7 years or less. These men had been transferred from camps

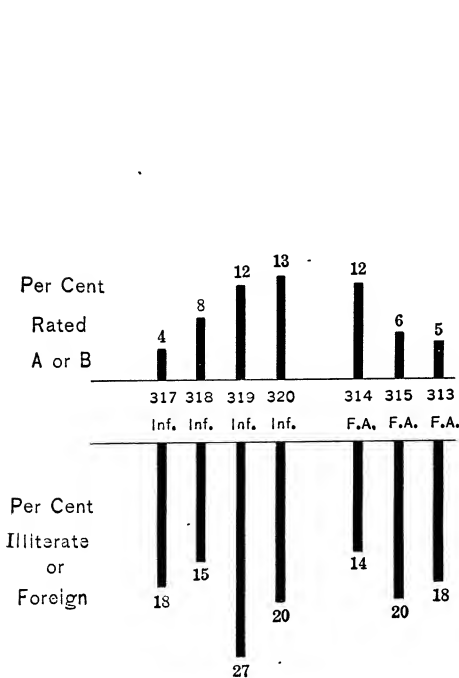


FIG. 12.

FIG. 12.—INEQUALITY OF REGIMENTS.

FIG. 13.—INEQUALITY OF MENTAL STRENGTH IN 18 OFFICERS' TRAINING SCHOOLS, 4TH SERIES (TOTAL ENROLLMENT 9,240).

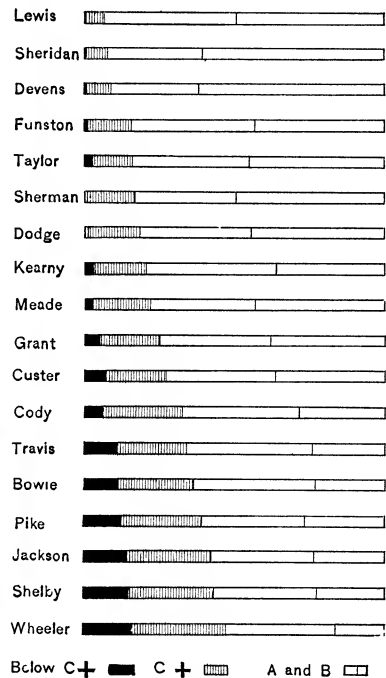


FIG. 13.

where there were no psychological examiners, consequently they had not been examined previously. Such data illustrate the danger incurred in building units without regard to mental ratings. See also Fig. 8.

In a unit about to go overseas 306 men were designated by their commanding officers as unfit for overseas service. These were referred for psychological examination, with the result that 90 per cent. were found to be mentally 10 years or lower, and 80 per cent. 9 years or lower, see Fig. 9.

Mental tests reveal the weak links in the army chain. As a result of findings like those illustrated by Figs. 10, 11, and 12, the intelligence ratings are being widely used as a basis for equalizing or balancing the

MENTAL TESTS IN INDUSTRY

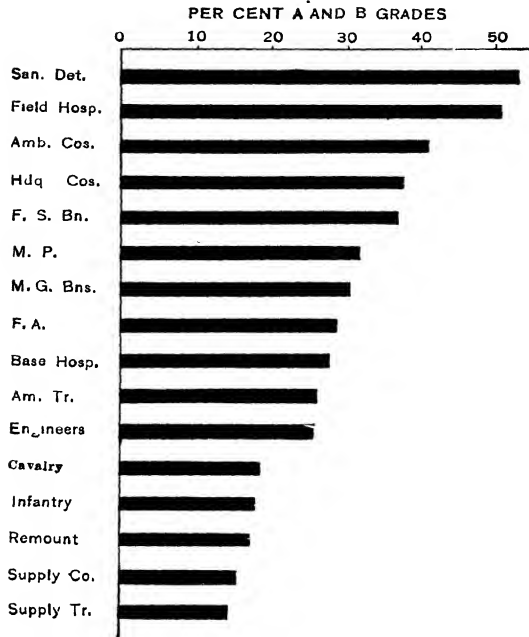


FIG. 14.—COMPARISON OF ARMS OF SERVICE.

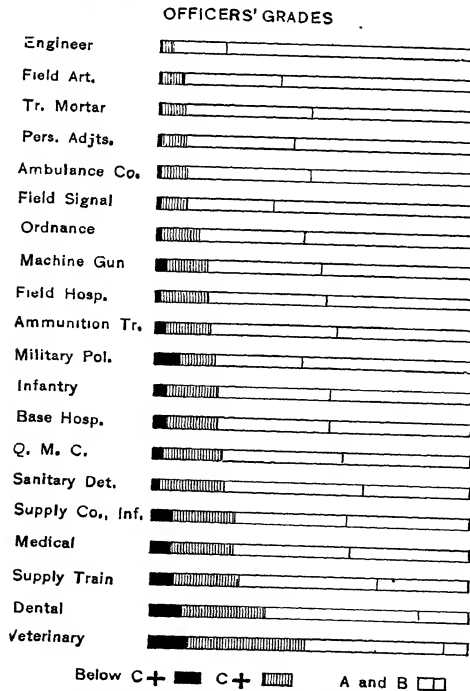


FIG. 15.—PROPORTION OF HIGH AND LOW GRADES IN VARIOUS OFFICER GROUPS.

mental strength of units. As many as 25,000 men have been permanently assigned to a division in a few hours on the basis of intelligence scores and tables of occupational needs. In the engineering regiment shown in Fig. 11, a redistribution of men was made on the basis of the evidence

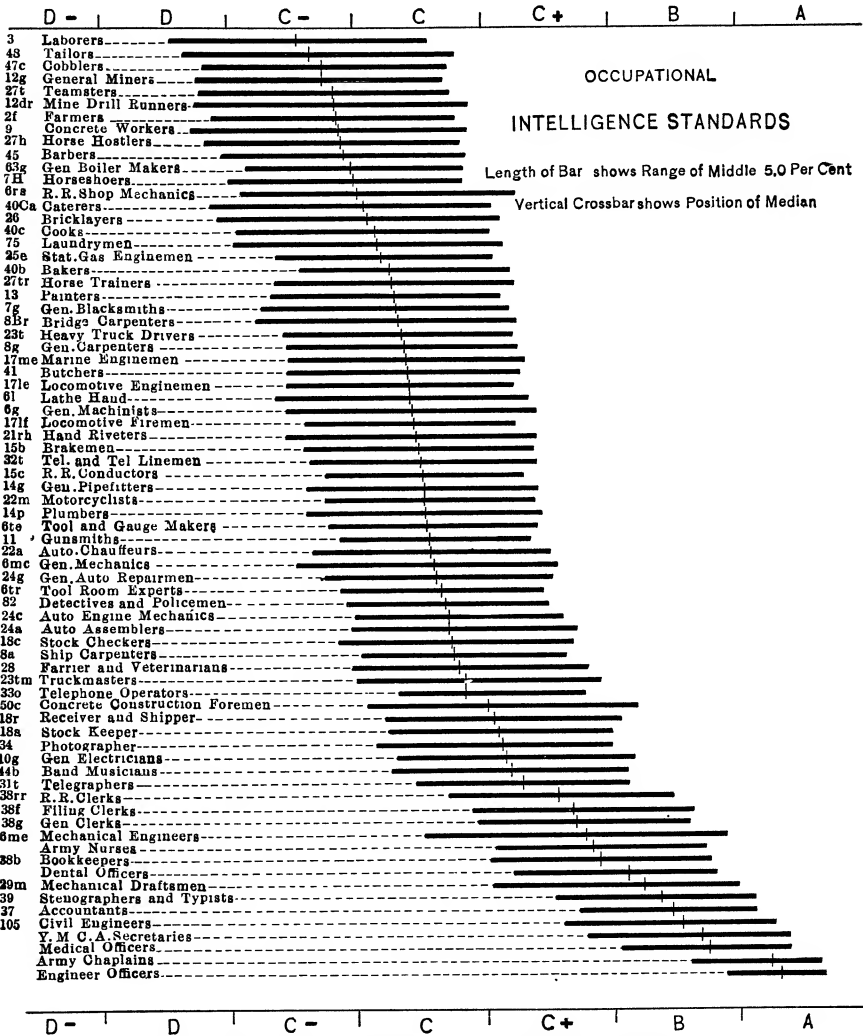


FIG. 16.—OCCUPATIONAL INTELLIGENCE STANDARDS.

submitted by the psychological examiners. One year later an officer of this regiment reported that, in the opinion of the officers of the regiment, its efficiency had been increased 100 per cent.

In replacement units interesting experiments in training are in progress. Often the A and B recruits are placed in one group, the C+,



C, and C— men in another group, and the D and D— men in a third group. By such classification, it has been found possible to secure greater speed in training. High-grade men are not delayed by the inapt, and low-grade recruits are given specially qualified instructors, special forms of drill, and various kinds of individual attention. Thus all recruits progress as rapidly as their individual ability permits.

The proportion of A grades in the schools shown in Fig. 13 varied from 16.6 to 62.4 per cent.; the proportion of A and B grades combined from 48.9 to 93.6 per cent.; and the proportion below C+ from 0 to 17.9 per cent.

Fig. 14 shows the per cent. of enlisted men grading A or B in various arms of the 34th division. Different arms of the service do not require the same mental strength. Experience shows that few men of D or D— grade can be safely used in field artillery, machine-gun battalions, or field signal battalions.

The proportion of A grades, in various officer groups, as shown in Fig. 15, varies from 8 to 79 per cent.; the proportion of A and B grades combined, from 52 to 96 per cent.; and the proportion below B, from 4 to 48 per cent. Note the remarkably high ratings of engineer officers.

In Fig. 16, the bar shows range of middle 50 per cent. The vertical cross bar shows position of median. The figure is based on data for approximately 36,500 men. Numbers at extreme left are key numbers of occupations. Data are taken from soldiers' qualification cards. These data, although inadequate for practical purposes and relating to the army alone, suggest the possibility of securing intelligence specifications for important groups of occupations.

## DISCUSSION

B. F. TILLSON,\* Franklin Furnace, N. J.—I would like to ask Major Yerkes if he will not give us more examples of the tests than are contained in the printed pamphlet. It would be of great interest to study them more carefully.

ROBERT M. YERKES, Washington, D. C.—The reason for not reproducing the methods in any public print is that it lessens their value and necessitates the preparation of new test materials.

BRADLEY STOUGHTON, New York, N. Y.—I hope that this matter will be of so much interest to the members of the Institute that the Directors will decide to appoint a committee which can bring further results of this practice before us. I understand that Columbia University, at least in some departments, has already adopted the Binet tests and is using them. I understand that some industries have considered seriously the question of grading their employees in accordance with

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\*Mining Engineer, New Jersey Zinc Co.

some mental tests. If that is to prove of value, the quicker the mining engineers and metallurgists are familiar with the proper people to carry on the work or the proper methods to be employed, the better. We generally work through committees, as you know, and I hope one may be established.

One thing that interested me was the high rating of the engineers and I questioned whether these mental tests were not rather more easy for engineers than for men of other vocations, although of equal intelligence. Engineers naturally are trained to mental impulse and that I believe is one of the characteristics of the different tests. I noted in particular that the telegraphers had a large number of high-grade men and a very small number of low-grade mental individuals. It occurred to me that also might have something to do with the occupation of the telegraphers. Any of you who have played with telegraphy know how instantly the application of an idea must follow the slightest variation, the ticking must be recorded immediately in the movements of the pencil to get down the message.

R. M. YERKES.—It was suggested to us early in this work that there was some special reason for the high rank of the engineers. We analyzed our results to find out whether the tests were easier for men with mathematical training. The evidence pretty thoroughly indicated that the engineers deserve the position which our tests give them.

It is interesting to know that in the army schools for chaplains, extremely high ratings appeared. We compared the chaplains' records with those of engineers and medical men and found that the groups had obtained their ratings in different ways. The chaplains were skilled in the use of languages and they scored heavily on the tests that depended largely on language, whereas the engineers were stronger on the reasoning tests.

B. F. TILLSON.—May I ask whether these tests showed whether a man were moral or honest and whether he was likely to go to sleep on the job?

R. M. YERKES.—The tests measure intelligence or mental alertness; nothing else.

## The New Spirit in Industrial Relations

BY HERBERT M. WILSON,\* PITTSBURGH, PA.

(New York Meeting, February, 1919)

WE of the employer class represent labor in the social organization and in industry just as truly as do those who labor only with their hands, and, because our labor is chiefly with our brains, the duty is all the more imposed upon us to use those gifts in which we excel in helping steer a clear course through the turbulent waters in which we are now enmeshed. Only their selfish engrossment in the daily tasks they were employed to perform could possibly have permitted the great majority of the intelligent and industrious German people to become terrorized by a handful of the idle, the vicious, and the criminal, whom the elections to the National Assembly showed to be in as pitiful a minority as they are in Russia, in this, and in other countries. It is the leisure unemployment of the I.W.W.'s, the Bolsheviks, and the Spartacists that has enabled them to devote their whole time to a concerted and aggressive struggle against the great majority of those of the world's workers who are unorganized, and who are therefore even in opposition one to the other. In union there is strength, and this was never better illustrated than by these conditions.

The great fallacy of our previous attitude toward social and industrial problems has been the belief that labor, by which we commonly mean organized or physical labor, is something antagonistic to and having a different object in life from that great body of unorganized laborers who work with their brains. Our difficulty has been our lack of will or opportunity to think. The hurry of our daily struggle to earn our livelihood under the nerve-racking pressure of modern civilization has left us no opportunity for quiet thought and study.

The physical laborer, and above all the laborer in the mines, on the other hand, has all his hours of toil in which to think over, in all their varied aspects, those problems that concern his immediate, personal well-being. As a result he has arrived at perhaps a more true appreciation of the facts of life in their relation to social and industrial conditions than we. Have we not too readily adopted the view that, as the managers of industry under the direction of capital, it has been our duty to think and act in opposition to labor, rather than to realize that we also are laborers, have like aspirations, and should participate in a free and aggressive solution of these problems rather than leave their solution

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\* Director of Department of Inspection and Safety, The Associated Companies.

to those who are taking the direction of affairs solely because of our indifference and misunderstanding?

No one has more clearly analyzed the situation from the point of view of the employers, and few have done a better service for industry, than John D. Rockefeller, Jr., in the courageous and enlightening addresses recently delivered by him on this subject in Denver and in Atlantic City. He has clearly shown that there are not two factors to this problem, as many had thought, viz., Labor and Capital; or three, as others have believed, Labor, Capital, and the Community; but four: Labor, the Community, Capital as represented by the stock and bond holders of industrial property, and the Management as represented by those collaborators employed by Capital. It was he, also, who first analyzed clearly these relationships and sought their solution a few years earlier, in consequence of the industrial warfare in which his properties, the Colorado Fuel & Iron Co., were then involved. As a result of his painstaking and open-minded investigation of the underlying causes of these troubles, he developed a reasonable and coöperative solution in what we now call the Colorado Industrial Plan. It is not only my belief, but evidently the conviction of many of the industrial leaders, that in some such plan lies the solution of our industrial and social problems.

No people in the world are better prepared to take the leadership in a right solution of world conditions than those of that great nation which, for a century and a half, has lived under truly democratic and representative government—the United States. That the whole world realizes this is evidenced by the leadership unanimously accorded at the International Peace Conference to President Woodrow Wilson, and the equally frank leadership accorded by the conservative element in labor to Samuel P. Gompers.

#### PROGRAMS OF LABOR LEADERS

While all know, how many have stopped to consider the tremendous influence actually being yielded by the representatives of physical labor, as well as by the representatives of unemployment, in the solution of the world problems now engrossing the world? These conditions have brought to the front four great international conferences that are in session at the same time as the International Peace Conference: The anarchistic and ultra-radical group recently called by Lenine to meet, wherever they may, their fellow Bolsheviks; the international Socialist and Labor conference in Switzerland, at which such radical labor leaders as Henderson, of Great Britain, and Thomas, of France, are meeting with the Socialists of their enemy countries, Germany and Austria; the less radical and anti-German Socialist group meeting in Paris under the leadership of Russell, of the United States; and the more conservative and

clear-thinking Labor group now meeting in Paris under the leadership of Gompers.

In view of the conditions thus briefly sketched, I want to record my conviction that it is our duty to our employers, to our associates, to ourselves, and to our successors to determine here and now to aggressively participate in securing a right solution of these problems, rather than to leave them to the less capable, if more earnest, manual laborers, or worse, to the Socialists or Bolshevists.

As nearly as I can now ascertain, the more conservative programs of the Labor and Socialist conferences now in session advocate the measures adopted at the conference held two years ago at Leeds, England. Among these are the 8-hr. day, anti-child-labor, anti-night-labor for women, proper hygienic conditions, and accident, sickness, old-age and unemployment insurance. The newly organized British Labor Party, representing a combination of the conservative and other labor groups with the political Socialists, has a representation of perhaps 100 in Parliament. Its program demands, among other things, an employment insurance, democratic control of the industrial methods and ownership with equitable share of proceeds, nationalization of electric power, mines, railways, telegraphs, and industrial insurance. Compared with this, the program of the spokesmen of American labor appears modest indeed. Their present extreme demand is for an 8-hr. day, the right of workers to bargain collectively, organization of all unskilled labor, and opportunity for intercourse and exchange of views between workers and managers. The Department of Labor emphasizes the importance of granting the last proposal. Charles Schwab has stated that labor is inevitably destined to share direction in the control of all industries, as have Judge Gary, John D. Rockefeller, Jr., and others. In view of this comparison, I think it a fair statement that the American Federation of Labor and the United Mine Workers are perhaps the most active and powerful influences in this country tending to divert working men from Socialistic theories to the more practical aspects of social life founded on our national ideas.

#### EVILS OF COMPULSORY INSURANCE

We can well add to the considerations that I am about to submit to you the following measures, providing they are shorn of certain Socialistic aspects. These latter are usually described by the title Social Insurance and are intended to be operated as a function of government. This aspect of compulsory insurance is distinctly German; it is wholly paternalistic or autocratic in its purpose, and is opposed to every fundamental principle of our democratic institutions. As Frederick L. Hoffman, America's most expert student of the statistics of accident and health has pointed out, the Imperial Government adopted social

insurance for the purpose of combating the growth of Socialism. Social insurance was expected to complete the subjection of the German wage earners to the will of autocracy. On the contrary, the spirit of Socialism has been greatly strengthened because compulsory social insurance rests upon a typically German misconception of the relations of labor and of civilization to democracy, for it involves the establishment of a permanent class distinction of a most insidious nature. In contrast to this European endeavor to better the condition of the wage earner by quasi-state charity, is the material improvement in social conditions in this country by the establishing of those health and welfare institutions which really serve better the purpose of state insurance; such, for instance, as more and better hospitals, infirmaries, and dispensaries; better safety-first laws and regulations; and better welfare activities.

Unfortunately, certain American publicists are sounding the praise of the German system of social insurance. A few of them have fallen into the trap of the late Imperial Chancellor, who planned what the Socialist conference in Switzerland is now proposing, namely, a provision in the treaty of peace for the insurance of labor. These political economists of ours have endeavored to introduce into one state after the other a system now thoroughly condemned by American labor leaders and the American medical profession, and not endorsed by any American business institution. Government and State insurance, and especially that type of insurance known as old-age, sickness, and unemployment, can only have the effect of making the workman a parasite of the state, a tool of political intrigue, and an instrument for the perpetuation of the party in power. We value only those things that cost us money and labor. Insurance, to be effective in establishing and maintaining the incapacitated and for the encouragement of those on whom they are dependent, should be managed by the private endeavor either of industry or of the insurance business.

#### ADVANTAGES OF GROUP AND COMPENSATION INSURANCE

Group insurance, a plan devised by American insurance agencies, will do much toward fostering desirable industrial conditions, because it is a well-known fact that assistance given in time of distress is much more appreciated than that given at any other time. The occasion of each death serves to renew in the minds of the employees the advantage of working for an employer who, in this respect, safeguards the welfare of his employees. By thus fostering these personal, human relations, and because of the continued reasserting of its advantages, group insurance is superior to most other forms of welfare work, the appreciation of which often wanes when the novelty has worn off.

Casualty insurance of the Workmen's Compensation obligation has, in like manner, been made by the stock insurance companies a companion measure for the protection of the workman and his dependents and for safeguarding him from accident. The effect of these forms of insurance on labor turnover, the elimination of the cost and frequent hardship attendant upon an employee's incapacitation or death, the general improvement in *esprit du corps*, and the marked drawing together of employer and employee have been remarked by all who have had to do with these measures of industrial welfare.

#### RELATION OF THESE QUESTIONS TO THE MINING INDUSTRY

And now to a more detailed discussion of the bearing of these questions upon industry, and, in particular, upon the mining industry. It is only in recent years that this problem has developed until it has taken on those aspects that are now clearly defined. But a score or more of years ago, labor and capital were intimately associated in the then small and independently owned industrial enterprises. The capitalist was the owner and manager of the industry and his employees were so few in number and lived so near their place of labor that they and the owner had ample opportunity for personal and intimate social and business intercourse. As a result there were fewer social problems to cause misunderstandings, strikes, or lock-outs. As President Wilson has well said, when men meet face to face in friendly discussion of their common needs, some satisfactory solution is sure to be found. Even yet, many of the smaller independent mining or other industrial operations are successfully handling these problems through the personal interest of the owner in the well-being of his employees.

As modern industry has developed, ownership has expanded to the control of huge and diversified industrial activities financed by the public under the title of Capital. In consequence there has grown up between labor and capital an administrative organization known as the Management, so that these two, Capital and Management, constitute an impersonal and distant substitute for the individual employer. It is this condition which has in recent years so seriously complicated the employment problem. The employer has allowed himself to be so engrossed with the financial, technical, and physical aspects of his business that he has been willing to muddle through his relations with labor as best he might. On the other hand, the laborer has been so engrossed in his endeavors to secure a livelihood under whatever living and working conditions might be offered him that he has given little thought to his relations to his employment, until some misunderstanding has ended in a strike. No thinking man doubts that were the machinery automatically available under such circumstances for friendly, personal,

face-to-face consideration of the problems confronting employer and employee, there would be fewer disagreements, fewer strikes, and a better and more settled industrial situation.

The necessity for some such medium for exchange of views has resulted in the organization of labor and, in some measure, of employers. Each party, engrossed only in defending its own viewpoint, has generally tried to gain its objectives by might rather than by right. Gradually, however, the representatives of both elements are beginning to appreciate that when the machinery for bringing them together in friendly communion is available their differences frequently settle themselves. There remain yet vast numbers of employers and of employees who are not available; for these the machinery should be provided.

Employment of labor may be considered under two headings: method of employment and method of retention. The manner of treating each of these divisions of the subject will depend on the size of the mining operation. The method of employment for a large operation may be by the maintenance of a separate employment department; for small mines, there may be no employment department or organized method of employment. The method of retention of labor will be effected by the size of the operation, the conditions of employment, its possible future permanency, the opportunity for promotion, and the general working and living conditions.

Employment by a large industrial operation should be through a separate department of labor and welfare, which should have liberal appropriations and establishment, as has E. L. Close's department of the United States Steel Corpn., and be governed by definite rules and methods. The hiring of labor should be governed by classification, a personal method of interview, and by consideration of the appropriate working place and other conditions that will affect the prospective employee. The employment and the retention of labor are affected not only by the questions of wage and chance of promotion, but by the conditions surrounding the work and the working place, and by those connected with living opportunities. No aspect of the method of retention of the right kind of employee is more important than that which concerns the living and recreational opportunities offered near the place of employment. These have been generally described under the titles of Safety Work and Welfare Work; the first deals with the place and conditions of the employment and the latter with the place and conditions of living.

A better knowledge of the effect of these considerations on labor and employment has convinced all employers and employees who have had personal experience in such matters that an improved condition of safety adds to the efficiency of mine operation and to the comfort and well-being of the miner; and that good living conditions, including housing, educational, and recreational facilities, and the security of



insurance, add in equal measure to the efficiency of the mine operation and to the comfort and well-being of the miner. Both of these so-called activities tend to reduce the cost of production; to increase the days of work, and hence the earning capacity, of the employees; and to make for the more permanent retention of labor, and consequently the preservation of a higher grade of labor.

The question of separation is often one of the most difficult and most full of opportunity for misunderstanding. When either the business conditions or the personality of the employee may require his dismissal, it is necessary to decide how this shall be done in such manner as to make clear not only to the employee but to his associates that his separation is necessary. A common method is by a brusque notice of discharge; a better way would be by giving notice of one or more weeks prior to compulsory dismissal; or better still by a personal interview with an opportunity for voluntary withdrawal within a fixed time.

A great deal of important and constructive work has been done toward the solution of employment and welfare problems by a hundred of the larger mining corporations scattered from the Atlantic to the Pacific, notable among which are the mining subsidiaries of the United States Steel Corp., the Republic Iron & Steel Co., Nevada Consolidated Copper Co., Utah Copper Co., Copper Queen Mining Co., Cleveland Cliffs Iron Mining Co., New Jersey Zinc Co., Consolidation Coal Co., and the Ellsworth Collieries Co.

The most promising, and one of the most successful recent efforts to solve this problem is that above referred to as the Colorado Plan. Its success as a means of bringing about better relations between employer and employees and in securing more permanent and better labor employment conditions appears to be amply testified to by the results of the past three years of successful operation. It is a striking example of the value of personal friendly conference, through the agency of a permanent establishment for the settlement of employees' troubles. In substance, it consists of a representative committee, selected by the miners and the owners, that meets at regular intervals to hear and inquire into the complaints of employees and the differences between employer and employee at each plant, respecting method of employment, retention, and separation. In the field of retention, this plan has adopted a most liberal attitude toward safe working conditions and satisfactory living conditions. It operates through committees on coöperation and conciliation; on safety and accidents; on sanitation, health, and housing; and on recreation and education. These committees have accomplished much for the betterment of the working and living conditions of the miners and their families.

A recent evidence of the trend of this new idea in the settlement of industrial disputes was furnished by the strike just settled by the General

Electric Co. through the management turning the whole question over to a Shop Committee and telling the members to get together and settle the disputes. The result was a prompt, and to all parties concerned, a satisfactory settlement, and resumption of work under conditions that promise reasonably permanent stability. An earlier illustration of this method in a semi-national way was the settlement of the anthracite strike of over a decade ago, through the appointment, by President Roosevelt, of the Anthracite Commission, representing employers, employees, and the public. As a result, labor, in the person of John Mitchell, was then thrust into that industrial and political prominence it has since held in increasing strength.

The latest step, and possibly the one of most serious portent in this regard, is the Whiteley Report of the British Ministry of Reconstruction. This report recommends the formation of Joint Industrial Councils—national, district, and works—for each of the various industries. Labor, capital, and the public are to be represented equally on each. More recently, the British Commission on Unrest has recommended to the Prime Minister the adoption of the principles of the Whiteley Report, with certain extensions.

What I have endeavored to present is in no sense a labor employment program. It is rather a review of the idealistic aspirations of those who have been forced to consider a problem so complex and so vital that it affects every phase of organized civilization. I may arouse in some the fear that the Colorado Plan or the Whiteley Report contemplate a dangerous participation by labor in the management of industrial enterprises—this is far indeed from my conception of their purposes. I would say rather that they propose only that one of the parties to industrial production, the man as distinguished from the machine, shall participate in the management of those affairs that chiefly concern him, namely, his working and living conditions.

I might epitomize all that I have said by the statement that in all industrial operation two types of machines are used, the mechanical and the human. Heretofore, and due in large measure to the teachings of our technical institutions of learning, all our education has been concentrated on perfecting the design, permanency, and repair of the machine; and, heretofore, our educational institutions have given nearly equal consideration to the perfection of the human machine, but a little thought to its permanency, and none whatever to its maintenance and repair. When worn out, it has gone to the scrap heap, shorn even of scrap value.

## DISCUSSION

W. D. BRENNAN,\* Cheyenne, Wyo. (written discussion†).—My experience has been that, where possible, it is preferable for each fore-

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\* Asst. Gen. Mgr., Union Pacific Coal Co. † Received Jan. 24, 1919.

man to employ his own men, rather than to have them handled through an employment bureau. The fact that each foreman comes into direct contact with the man before he is put to work gives him an opportunity to choose, at certain times, the kind and class of men that he wishes. Where possible, after a man is employed he is sent to a central office where a complete history of each individual is kept on a card. If a man has previously been in the employ of the company, his history can be obtained from this card, and if for any reason he is not wanted he can be refused work at the time he reports to the central office.

In connection with Workman's Compensation costs, we in Wyoming feel that although the rates of compensation paid may in some cases be rather low our law works satisfactorily. As the state and the number of employees are small, compensation cases are handled through Judges of the various District Courts. In the State Treasurer's Department, a few clerks were added to look after the handling and issuing of warrants and compiling of statistics. Most of the large employers have an arrangement by which a joint claim and assent, specifying the amount of compensation due, is made up by the employer and signed by the workman. This is forwarded to the Judges so that personal investigation of individual cases is not necessary. In this manner, compensation is promptly received and overhead expenses of operation of the law is kept at the minimum.

The law requires each employer to pay into the State Treasury, for the benefit of the industrial fund, a sum equal to 1.5 per cent. of the money earned by each employee, engaged during each calendar month of such employment. Each employer contributes monthly, unless the sum heretofore contributed, after deducting all payments made on account of injuries to his employees, shall equal a full 1.5 per cent. of his annual pay roll, computed by multiplying his current monthly pay roll by twelve, and shall likewise be not less than (\$5000). It has been found during the years that this law has been in effect, that a number of the larger employers have been exempt from payment for from five to eight months per year, so that in some cases, even in coal mines, industrial insurance has cost less than 0.75 per cent.

The question of relationship between employer and employees is one where personal friendship is of the utmost importance. We of the West probably have, or at least feel that we have, a greater freedom than exists in some other parts of the country, so that we possibly come in closer contact with the employees than is possible in some other localities. This, in a number of cases, has been the means of avoiding serious trouble. The closer the personal relations between the employer and the employee, the easier it is to get together and talk over difficulties and questions that are constantly arising. The fact that the employees are allowed to state their grievances and have an opportunity to be shown the employers'

point of view will, in many cases, go a long way toward keeping relations friendly.

WILL L. CLARK, Riverside, Cal. (written discussion\*).—In the large operating metal mines of the western part of the United States the situation is one of much complexity, due to the plan of wage schedules effective during the European war and based on the average monthly price per pound of copper. It would have been much better for the western mining industry, in general, if an allotment had been made from surplus annual earnings, for it seems most desirable that workmen should have some interest in an operating company experiencing a good year and producing an amount sufficient to allot each workman, having a clear record, \$100, or more, annually.

Detailed attention to welfare provisions is now the practice, in all western mining camps of the large copper companies. The question of uniform wage schedules should be formulated promptly; and during its consideration with employees, there might be included the preliminary discussion of uniform workmen's compensation plan, including sickness and accident allowances. It is a serious reflection on the ability of the executives of western metal mining companies that they have not devoted time and concerted effort to reach an agreement with employees as to a sensible wage schedule and compensation for sickness and accident; but instead have run along under a haphazard temporizing policy. This necessity once met and arranged for, the mining camps as now conducted, free from the saloon evil, offer high wages and fine climatic and, generally, good living conditions.

The best opportunities for investments, where one is able to form first-hand opinion, are producing mines or speculative chances in new prospects being opened in a miner-like and proper business manner, as differentiated from the wild-cat, stock-selling promotions, also the products from nearby farm lands are assured of an exceptional market and many miners invest and retire to country life.

This digression from the main subject, which has been well stated by Mr. Wilson, is to call attention to the opportunity that exists for young Americans in mining and related occupations. The metal mines and ore-reduction plants offer work that is interesting and not arduous. The young Americans appear recently to have taken up mining work, not as a vocation but as an opportunity to travel, jumping from place to place, while the foreigner, appreciating good wages, has held on, until the proportion now engaged is surprisingly large, in some mines 80 per cent. An observer returning from the battle front in France said there were three things noticeable about the American soldiers, in camp: First, they were regularly hungry; second, broke; third, always wishing they were

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\* Received Jan. 31, 1919.

somewhere else. The last is true of our young men with reference to all ordinary or common labor. A large proportion of the orchard and garden workers in California are Japanese, Chinese, Portuguese, Mexicans, and a few Indians and other native Americans. American youths have not displayed sufficient persistence to make producing war gardens. Can they be induced to take up mining and other ordinary vocations and, if so, how is efficient work to become the rule and provide for the continuance of high wages? In a recent article, Mr. Mitke says "Time studies will lead to mutually advantageous results, but in the minds of workmen, unfortunately, the idea of time studies is so closely associated with speeding up the work that they are inclined to look on them with suspicion."

One of the reasons for this belief is that the men are not informed and given opportunity to coöperate in the whole study. Clearly a better understanding with the workmen can be arranged and every one, by meetings, lectures, and examples, shown the advantage to him, and his class, of accomplishing a fair day's work, without waste of effort. Every local superintendent and manager knows how almost maddening it has been to observe unskilled and time-killing laborers, and to feel, since the foreman or boss is no longer a driver, that there was not available any relief. Now the immediate necessity of bettering the labor situation should evolve this and conferences be arranged to formulate agreements.

Another phase, which Bolshevism has made immediate, is brought out in the annual report of the Secretary of the Interior, Hon. Franklin K. Lane, for 1918. He says: "To the necessity for more thorough education of the people all countries have become keenly alive. As we move further and further from the war, we will discover much that we do not now see. But this one thing stands out more plainly than ever before, that this world is to belong to the workers, those who do and those who direct the doing. Not merely to those who drive the nail or lay the brick, but also to those who have come to a higher capacity through education and large experience, the men of scientific knowledge, of skill in the arts, of large organizing capacity, ease, and sheltered repose will come only to those who themselves have earned it." In the opinion of the writer, it is of the greatest moment that wage schedules for metal mines be formulated and all the time and effort required be devoted to the adjustment to practical working and living conditions.

C. W. GOODALE,\* Butte, Mont.—In regard to the employment manager, the North Butte Mining Co. has undertaken that line of work and the rest of the companies are watching the experiment. If it is proved to be the proper thing, the system will be introduced into other mines. We have to combat the opposition to the use of the "rustling" card.

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\* Manager, B. & M. Dept., Anaconda Copper Mining Co.

It is nothing more nor less than asking the nationality, where the man formerly worked, and whether he is married or single.

ROBERT RHEA GOODRICH, Anaconda, Mont. (written discussion\*).—Since the efficiency of the different phases of personnel work employed by industrial concerns, which are bringing about greater efficiency and lessening the labor turnover, the latter being intimately connected with "contentment of mind," depends on the individual workman, it is highly desirable, as far as industry is concerned, to keep the workman's mind and body in trim in order to lessen accidents and produce a higher grade of work. Prevention being better than cure, health supervision of the workmen during employment should be maintained by the employers in order that mental alertness and physical well-being may be obtained. I have long believed, from observation in many plants, that when any problem is dependent on the individual workman, what he believes and what he does is vitally dependent on his health. I will follow with much interest what is to be done in the future in relation to the subject of health supervision during employment (as well as medical examinations at the time of employment), and I hope this idea will be generally instituted in personnel work.

A workman with good health and a contented mind should be an ideal component of a progressive industrial concern. The contentment of the workman is fostered by a number of admirable phases of welfare work which are being still further developed. The mental and physical well-being of labor should be the issue in Labor vs. Capital in the new period of the industrial promotion of the United States into which we are now entering.

M. D. COOPER,† Brownsville, Pa.—In the bituminous mines the whole system at the present time, with a few notable exceptions, is lost in confusion. In fact, there is no system. At the average mine, the superintendent or foreman employs his men with very little examination of any kind, physical or mental. There is need, therefore, of some standardization of methods. This is accomplished, in the case of the larger mines, by an employment bureau, but in western Pennsylvania there are a great many mines, employing from five up to 100 men, which are at a disadvantage when it comes to maintaining any sort of employment bureau. But it is possible to establish a system without an employment department; that is to say, some form of a record may be made of the men and their fitness for certain positions and this record passed about among the employees in the handling of the labor situation. A better method would be a coöperative employment bureau among the small employers.

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\*Received Feb. 17, 1919.

†Assistant General Manager, Hecla Coal & Coke Co., Pittsburgh, Pa.

The next feature of the employment of mine labor is that of retention. This, during the past two years, has been an exceedingly serious problem. Two factors affect the retention of mine labor: The first is the reputation of the employer. This is dependent largely on living conditions; that is the location of the plant, the character of the houses provided, sanitation, etc. Recreation is increasingly important. The company with which I am associated has recently attempted to solve the problem of recreation and add to the contentment of the community by erecting a community house, which contains a moving-picture show, bowling alleys, pool tables, etc. This is a move in the right direction. Education also is an important feature. The miners are much more interested than formerly in the educational facilities provided for their children. Next, after the reputation of the employer, is the conduct of the bosses in the company organization. It has been the constant effort of our company to obtain for the official positions men who were able to take a broad-minded view in regard to mine labor. This is not an easy thing to bring about, but on its accomplishment depends the success of the company in retaining men. In this connection the point Dr. Lanza has brought out in regard to the examination of men is very important. I suppose we can all think of men who are employed in certain positions where they are entirely out of place physically.

The third feature of employment of mine labor is that of disconnection with the company; there is vast room for improvement here. The solution of this problem lies in the giving of sufficient notice both by the laborer when he intends to quit and by the official when he intends to discharge the man.

THE CHAIRMAN (B. F. TILLSON,\* Franklin, N. J.).—It seems to me that the employment of labor is so closely knit with the problem of labor turnover that it would be of great interest if our *Transactions* contained data, or figures, from representatives of various companies in regard to what their labor turnover has been during this past year. I may say that the zinc mine at Franklin had a labor turnover of 72 per cent. for the past year. Furthermore, the question of attendance enters importantly in production. If the attendance is poor and the foreman does not know on how many he can rely, it produces inefficiency. The attendance during the same period was 87.5 per cent. of the possible shifts of labor, which includes two months' suffering from the epidemic of influenza, which was very severe in the camp. The lack of attendance may be classified as follows: Loss of time due to accident, loss of time due to illness, loss of time from other causes, realizing that the loss of time due to illness is often brought about by inebriety. The corresponding statistics were for this same force of men, averaging about 850: Loss

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\* Mining Engineer, New Jersey Zinc Co.

of time due to accident, 0.5 per cent. of the total possible time; loss of time due to claimed illness, 3 per cent.; loss of time from other causes, 9 per cent., which indicates that a man's lack of interest in earning more and attending to his job are an important factor in the amount of work being done by the present forces of labor.

E. A. HOLBROOK,\* Urbana, Ill.—In listening to the remarks of the Chairman on labor turnover, the question arose in my mind, on what do we base our figures when computing our labor turnover? In one case, I was told, the labor turnover is figured on the basis of men working a month or more; in another place, the labor turnover is figured on the basis of a man going into his working place and putting in a shift; in another place, a man is simply put on the books of the company and is figured in the turnover. I would like to ask what is the basis on which you figure your labor turnover?

CHAIRMAN TILLSON.—The labor turnover is based on the number of men leaving divided by the average possible number of men that might have been working during the year, and the average possible force is obtained by dividing the total possible number of labor shifts by the average number of working shifts during the year. We have studied the problems from many angles and it seems to be a well accepted conclusion that this is the proper rating of labor turnover.

JAMES P. MUNROE,† Washington, D. C.—May I say, in connection with the very interesting paper that Mr. Wilson has presented, that I bring very encouraging news from the city of Lawrence, where my factory is. Lawrence has a very mixed foreign population so the Bolsheviki have been concentrating on it as the opening place for their campaign. For many weeks they have been sending their best leaders into the city to head this outbreak; but the movement has petered out. The foreign people have taken no stock whatever in these doctrines, with the result that all these leaders have gone away disgusted, saying that it is absolutely impossible to make any progress.

CHARLES F. WILLIS,‡ Bisbee, Ariz. (written discussion§).—An employment department operated only with the object of relieving the foremen from hiring and for the purpose of centralizing record-keeping is not likely to be entirely successful, and is doing but a small part of the good that practice has shown is possible; although it was probably a desire to save time and to conduct the business more con-

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\*Supervising Mine Engineer and Metallurgist, U. S. Bureau of Mines.

† Vice-chairman, Federal Board for Vocational Education.

‡ Consulting Supervisor, Dept. of Industrial Relations, Phelps-Dodge Corp'n.

§ Received Apr. 15, 1919.



veniently that led many mining companies to centralize their employment department. The ultimate object, however, is the reduction of labor turnover, by obtaining men better qualified to fill the positions to which they are assigned, by following up their work and providing for transfer to other departments or for promotion, if advisable, by reducing the number of quits for inconsequential reasons, and by supervising such laying-off as is necessary in such a way that the permanent force is not upset.

It provides a stability of employment, free from favoritism and paternalism, probably the best form of employment insurance that we can have. The value of the employment department is in direct proportion to the breadth of its supervisor, his ability to estimate and place men, his sympathy in dealing with their petty troubles, and his analysis of quits and the reasons therefor. There is probably no better index of the soundness of the industrial relationship between the employee and the employer than the reasons why men quit; an intelligent, unbiased analysis of these reasons tells where corrective measures are necessary. In nine cases out of ten the reasons are small grievances, easily corrected, and are things usually overlooked by mine officials.

An employment department has an immense field in the mining industry that has not yet been developed. In combination with the physical examination previous to employment, it permits of the proper placing of physically deficient men where they can do a day's work without injury to themselves or their fellow workers, it permits of the placing of war cripples, while it would be impossible to expect to find the twenty-five, fifty or one hundred bosses, whose motto is "rock in the box," broad enough to analyze the physical and mental needs of the various positions under them and select men accordingly.

Industry has no right to select its force of entirely physically and mentally fit men and throw into the market hundreds of thousands below that standard; each must take its share of the disabled and place them where their disability is not injurious to themselves, to the industry, or to their fellow workmen. It is only through centralized employment, the study of the problem by a man who is not required to show tonnage, that this can be accomplished.

## Prevention of Illness Among Employees in Mines

BY A. J. LANZA,\* M. D., WASHINGTON, D. C.

(New York Meeting, February, 1919)

THE prevention of illness among the employees of the mining industry is especially important in view of the importance of the industry, the unsettled conditions of labor, which emphasize the economic necessity for conservation of labor, and for humanitarian reasons. The burden of chronic illness is altogether unwarranted and unnecessary. If the industry is to maintain its labor supply in an efficient manner and keep the standard high, it is essential that it make every effort to keep its labor in good physical condition.

The basic principles underlying health conservation are the same for any industry, but they are here applied especially to the mining industry, and more particularly, perhaps, to metal mining, as it is with this phase of the industry that the writer is most familiar, and as it is conceded that metal mining presents more health hazards than coal mining.

The first step in the prevention of illness lies in securing employees who are in sound health and free from organic disease. I do not mean that all men who go underground should be physically perfect, but they should be free from organic defects of the heart, lungs, and other organs, or from anatomical defects that would markedly increase their liability to accident. The number of men working underground in mines in this country whose physical condition totally unfits them for such work presents a situation that should not be allowed to continue, and which I believe is not equalled in any other industry in the country. There is but one way to secure men who come up to the required standard, and that is by a thorough physical examination before employment. I am fully aware of the objections felt toward such a procedure by many employers and employees. There is much to be said on both sides, but to any one who is at all familiar with the mining industry as it is today in this country, and who approaches the matter with an unbiased mind, there can be absolutely no doubt as to the necessity for physical examination. As a purely logical proposition this is self-evident, but there remains the further necessity of placing physical examinations on such a basis that they can be administered in a just and equitable manner free from the abuses and suspicions to which they have been subject in the past.

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\* U. S. Public Health Service. Chief, Division Industrial Hygiene and Medicine, Working Conditions Service, Dept. of Labor.

The best way of administering physical examinations in the mining industry would be to have them conducted by properly constituted authorities of the various states. That is, if John Jones wished to work as a miner, when he appeared at the employment office of the Smith Mining Co., he would have in his possession a certificate from the state authorities declaring him to be free from organic or other defects that should, in the natural order of events, bar him from underground work. Such a system would presuppose reciprocity among the mining states and a basic standard of physical fitness. It would be well within the province of the Federal Government to establish such a standard for adoption by the various states. This arrangement may seem idealistic and impractical, and it is offered here largely for the purpose of causing reflection and argument, but at the same time, unless the mining industry can put into effect a system that will be fair both to employer and employee and that will prevent obviously unfit men from working underground, it will be almost useless to make any other effort toward prevention of illness among employees. I believe that the time has come for the American Institute of Mining Engineers to consider seriously the necessity and the means of carrying out physical examinations and to take a definite stand.

The second requisite for the prevention of illness among employees is the maintenance of working conditions underground on a high plane of sanitation and efficiency so that illness arising from working conditions may be held to a decent minimum. The greatest need in the improvement of underground conditions is the elimination of dry drilling in hard-rock mines. The relationship between hard-rock dust and miners' consumption has been so clearly demonstrated that it is not subject to argument; suffice it to say that the efforts made in England and in British colonial possessions to deal with this evil have shown the possibilities of abating it, while in this country little has been attempted. As long as dry drilling is permitted in hard-rock mines, just so long will we have an inordinate amount of pulmonary disease among the miners. That this has worked an economic hardship on the industry, aside from its humanitarian features, and that it has been one of the causes of the better grades of mine labor leaving the industry, is apparent to any one familiar with the conditions, and it is hard to understand why the subject of dry drilling has received so little attention in this country. Dry-drilling apparatus may be as deadly as a machine gun, if somewhat slower, and its continued use in the mining industry is a matter of reproach. While it may be true that the water varieties are not as satisfactory as they might be, at least their improvement should receive serious consideration. It is incredible that American ingenuity, which has produced the telephone, the telegraph, and the flying machine, should be unable to cope with the production of a water drill that would largely make impossible miners' consumption.

Conditions of ventilation and temperature underground are not what they should be and have never received the attention in metal mines that they have in coal mines. This is also a matter of prime economic importance because a greater amount of work can be done under conditions of proper ventilation and temperature than when there is poor ventilation and high temperature. While the latter may not cause specific illness, certainly they lower the vitality, lessen working ability, and predispose to many illnesses; if we are to have healthy miners we must so arrange it that they can work in places properly ventilated and not excessively hot. Mine ventilation is too complex a subject to be considered here, but the use of small fans with canvas tubing seems to offer a satisfactory and economical means of improving ventilation.

Proper toilet facilities underground are important as a health measure but vary so much according to the size and locality of the mine that they need not be gone into in detail here; besides, their principles are well understood.

The third requisite consists of adequate provision for medical and surgical service so that minor illnesses and injuries may be promptly treated and not become of major importance. In regard to accidents, "safety first" has become highly specialized and efficient; health service, if I may use this term, has not been developed to the same proportion. While many of the larger mining companies maintain first-class hospitals, the prevention of sickness among miners has not kept pace with the prevention of injury. It is just as feasible to distribute posters and other educational matter dealing with the proper care of common colds, constipation, and other minor troubles, which are forerunners of serious ailments, as it is to impress on the miners the necessity of having the smallest injury promptly attended to by the company doctor. There is need for a closer coöperation between the doctors and the miners in handling the minor ailments when they first appear. In former days the miner was an English-speaking person with all the advantages of a knowledge of the country and its institutions; at present underground workers are largely foreigners, and the employer must provide for their health during working hours and often in their homes if he desires to keep them on the job.

It is probable that the mining industry will always be hazardous to health and life as compared with other industries but at present it is needlessly so, because the wastage of human material can be prevented by securing physically fit labor, and by keeping its health unimpaired.

#### DISCUSSION

J. J. CARRIGAN, Butte, Mont. (written discussion\*).—I think all who have had experience in the operation of metal mines will agree with

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\* Received Feb. 17, 1919.

Dr. Lanza that many improvements can be made in the underground workings to improve health conditions. Physical examination would no doubt be a good thing and would do a great deal in abating the diseases now so common among metal miners, but I cannot see how it would be possible to put this examination into practical working order. Dr. Lanza says, "For the sake of argument, this physical examination should be under state supervision." I think if this were attempted it would result in nothing but argument. While such a law possibly could be passed in a few states, I feel certain that in the greater number the miners would fight such legislation with the same determination that they would fight a change of the working day from 8 to 10 hr. In the smaller mining camps this examination could probably be carried out under the supervision of the mining company with considerable success. In the large mining camps of the West that have adopted it, we all know how far it is from being satisfactory; it causes more discontent among the men than any other change of conditions that the companies may try to bring about.

Dry drilling, no doubt, has been one of the chief causes of tuberculosis among miners. While this practice is still being carried on to some extent, the reason for it is that there has been no wet drill that was at all satisfactory for use in all conditions of stoping. For some time, however, there has been on the market a spray used in connection with drilling that eliminates the dust in a satisfactory manner, but I know that 95 per cent. of the miners would refuse to use the spray and would sooner "eat the dust" than work under wet or muddy conditions.

The Anaconda company has been experimenting with a water stoper that, I think, will prove satisfactory. All the drill manufacturers have been working along these lines for some time, and probably the dry stoper drills will soon be entirely replaced by the wet drill.

Ventilation of metal mines is about the most important and one of the biggest problems to be considered, not only from a standpoint of improving health conditions, but as a matter of efficiency; but to compare the problems in the ventilation of coal mines with those of metal mines is hardly fair. The nature of the underground workings is entirely different, and the character of the ground in the metal mines is such that large amounts of timber are necessary, the oxidation of which generates a great deal of heat. By working on different levels in the metal mines the men are distributed over a large area, and there may be many different veins to be worked from each level, which makes it a great deal harder to control the air currents.

Until a few years ago, the mines in the camps of the West were operated by many small independent companies and the larger companies did not work together as they do now. Very little attention was paid to ventilation, at first, it not being necessary, and as the mines increased in

depth, these independent companies were lax in matters of ventilation. Since these companies have been consolidated, it has required a great deal of work to improve conditions, owing to the condition of the old workings and to the different elevations of the levels.

The Anaconda company has been going into the ventilation question very thoroughly for some time and has an engineer in charge of this work. I think we all realize what a big job it is and that it will be some time before the ventilation conditions are as the company desires to have them.

Regarding toilet conditions underground, all the mines of the Anaconda company are equipped with sanitary toilet cars, which are taken to the surface every day to be cleaned. I suppose that most of the companies in the country have similar equipment, as it is, no doubt, very important from a standpoint of health.

C. E. CALVERT, Butte, Mont. (written discussion\*).—Dr. Lanza's suggestion of an inter-state medical examination will meet with considerable dissension. It is safe to say that 50 per cent. of the miners are migratory, working from one camp to another and covering territory from Alaska to Mexico. These miners would object very strenuously to medical examinations at every move, and yet a medical passport between states does not sound feasible.

A law compelling medical examination would necessarily require some standard of health, and this also would cause dissension. Suppose an applicant failed to pass this standard, his medical card would show this and he would immediately become an undesirable, from the standpoint of an efficient laborer. If unable to procure employment on account of his health, he would naturally seek retribution from his former employer and one can readily see the difficulties in store should such a proceeding be carried out.

It is agreed by all that dry drilling is one of the chief causes of silicosis so prevalent among miners, yet this cause could be practically removed by proper legislation and strict enforcement of the law. Wet drilling in upper holes has not been developed because wet stoping machines have not proved entirely satisfactory. This lack of development by the companies may be attributed in part to the indifference of the miner. Had the miner demanded such a machine from the mining companies, they, in turn, would have demanded some improvement from the machinery companies. A few wet stopers have been tried in this locality but the personal element enters into the operation of the machine to such an extent that its success or failure depends on the individual miner.

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\* Received Feb. 17, 1919.

C. W. GOODALE,\* Butte, Mont.—A few years ago the miners made an unsuccessful effort in the legislature to have a law passed compelling the mine companies to furnish 100 ft. of free air per minute, exclusive of any air that was furnished from the air compresses, or the air used in drilling. At the present time the Anaconda company has machinery furnishing air at the rate of 300 cu. ft. per min. and it will soon be doubled, at the cost of \$500,000.

THE CHAIRMAN (B. F. TILLSON,† Franklin, N. J.).—I would like to ask Dr. Lanza if he would qualify his statement in regard to all rock being injurious by including non-siliceous rock?

A. J. LANZA.—I should say that if it was pure limestone it would not be considered contributory to tuberculosis. I mentioned hard rock. Some lime rock is siliceous and is injurious.

CHAIRMAN TILLSON.—Might I ask also whether that includes coal dust?

A. J. LANZA.—The question of coal dust is a very interesting one and is not entirely settled. There is considerable evidence to show that the coal miner is relatively immune to tuberculosis, and some very interesting views and arguments and demonstrations have been worked out by anatomists and pathologists as to why it is that a man who accumulates coal dust in his lungs is immune to tuberculosis. There is, however, I understand a growing notion that the dust may not be as harmless as supposed and I recently came in contact with men who complained bitterly of coke dust; but I have had no opportunity of finding out if it was doing them any harm. They complained that it was.

H. M. WILSON,‡ Pittsburgh, Pa.—Is it not possible that the immunity of coal miners to tuberculosis is due to the well known fact that one of the greatest preventatives and curatives of tuberculosis is the oxygen that comes from open-air life and plenty of fresh air; and therefore may not this immunity be due to the fact that in coal mines there is generally good ventilation and plenty of fresh air blown into the mine, and the men working in the mine are actually living under better conditions of good oxygen to breathe than the men in almost any other occupation that I know of?

A. J. LANZA.—I do not think that that is at all tenable. There is no getting around the fact that the inhalation of coal dust causes very marked changes in the structure of the lung, and it is also a well known

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‡ Director of Department of Inspection and Safety, The Associated Companies.

fact that the process of producing fibrous tissue is an offset to tuberculosis, as it does not afford a very advantageous ground for the development of the tuberculosis bacillus. As far as we can see, that is the only explanation that will hold water as to why coal miners are relatively immune to tuberculosis.

I want to say another word about dry drilling and about the remarks I made about dry drilling. I know perfectly well that the water drill was invented a good many years ago and have seen various water stopers and water drills and the like and all of us have seen water sprays in use on drills and we know these latter are largely worthless. There never was a water spray affixed to a drill that was worth the time and trouble it took to put it in place. There is only one kind of water drill that will be any good and that is a drill where water has to be used for the drill to function properly. We know that there are good water drills yet the dry drills are used in great numbers. There is absolutely no reason for that practice and you are not going to have anything but tuberculous miners while it is continued. There are all kinds of laws to prevent dry drilling; it is a question of enforcing the law. The point I want to make is this: dry drilling is practised to an inexcusable extent and I think the American Institute of Mining Engineers could very well take some steps to eliminate such practice.

CHARLES F. WILLIS,\* Bisbee, Ariz.—(written discussion†).—The consensus of the discussion of the paper of Dr. Lanza indicates that the physical examination, while it is probably a good thing, is fought by the workingman, and that, because of this fact, it has been proved unsuccessful. The physical examination standing alone is worth but little, and could easily be made a subject both of ridicule and dissension unless the company having such examination shows its own good faith in promoting a thoroughly better health program.

It is easily seen why a workman would resent his employer expecting him to be physically fit, if at the same time the employer provides unhealthful, unsanitary, working conditions, or if the town in which the employees are expected to live is dirty and unkempt. One mocks the other. The employer cannot say, "You must remain healthy so that I can get by with unhealthy conditions." But if working conditions are as good as can be obtained to keep men well, the employer can ask his men to be in a healthy condition.

A state examination and standard would have many difficulties, owing to the fact that a man would pass or would not pass the standard set, thus eliminating the discretion on the part of the employer in placing men whose physical condition might well permit working in one place and

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\* Consulting Supervisor, Dept. of Industrial Relations, Phelps Dodge Corp'n.

† Received Apr. 15, 1919.



not in another. Any physical examination that is run for the purpose of eliminating the physically unfit is unjust, both to the worker and to the nation as a whole; a state examination would make a distinct dividing line, whereas examination by the employer is conducted with a vision of the positions available.

The workman's opposition to the physical examination comes from the fact that he has been taught to believe that it is an interference in his personal affairs, that he can be clean or dirty as he chooses, that he can be healthy or unhealthy, and that it is nobody's business but his own. The war has taught us, however, that the individual is no longer the unit and that all such propositions must be based on the greatest good for the greatest number.

The opinion of workmen regarding the physical examination has changed within the last year, with the change of attitude of Samuel Gompers. As chairman of the Committee on Labor, he recommended to William B. Wilson, Secretary of the Department of Labor, that medical examination of applicants be made one of the functions of the government labor recruiting agency, reported in the Government Official Bulletin of Aug. 15. This recommendation was the outcome of a conference held in New York on July 15, and embodies the consensus of opinion by experts on the subject representing the laborers, industrial physicians, and public-health workers. The resolution stated that it was the sense of the conference that a physical examination of workers is primarily a measure of health conservation and also essential to maximum production, a war necessity, in that the purpose of a medical examination is not to eliminate the worker from industrial service, but to adapt him to the work for which he is physically fitted.

The standard examination card appears to conform closely with the examination card that has been in use for some years by some of the mining companies of Arizona, including the Copper Queen Branch of the Phelps Dodge Corp., the Calumet & Arizona Mining Co., and the Shattuck Arizona Mining Co., all operating in the Warren District. It deals with such subjects as laboratory diagnosis, general deformities, malnutrition and glands, diseases of the eye, ear, nose, throat, lungs, heart, nerves, and such general matters as hernia, hemorrhoids, and evidences of acute venereal diseases.

Indicating the possibilities in the plan, Mr. Gompers said: "There can be no question as to the wonderful effects such a movement as here proposed will bring about. It is only a matter of time and education before employers throughout the country will come under the rule that the people generally should be examined physically, and that the examination should be standardized."

The experience of the companies of the Warren District with the physical examination is that it has without a doubt led to very much im-

proved conditions of health, fewer accidents, and a general improvement in the physical caliber of workmen, due to the fact that curative methods are maintained and applicants for work can be made physically better men by doing work suited to their physical condition. The purpose of the examination of these companies, as stated by Mr. Gompers to be the right one, is not the elimination of the unfit, but the placing of men in positions where they can do best for themselves and for the company. The fact that the Copper Queen rejected but 68 men out of 2300 applicants, in a 6-mo. period, indicates this very clearly.

## Need for Vocational Schools in Mining Communities

BY J. C. WRIGHT, \* M. S., WASHINGTON, D. C.

(New York Meeting, February, 1919)

A PRACTICAL program of education for workers of the mining industries is being formulated by the Federal Board for Vocational Education in coöperation with the states in which this industry is a dominant one. The Federal Government last year passed a law, known as the Federal Vocational Education Act, that provides for the promotion of vocational education and for coöperation with the states in the promotion of such education in agriculture and trades and industries and in the preparation of teachers of vocational subjects.

More than 1,000,000 men in the continental United States are employed in mines and quarries. The following table, from the 1910 Census Report, shows the relative importance of mining and manufacturing pursuits:

	MANUFACTURES	MINES AND QUARRIES
Employees .....	7,405,313	1,109,410
Capital .....	\$18,428,269,706	\$3,380,525,841
Value of product.....	\$20,672,051,870	\$1,238,410,322

Of the more than 1,000,000 men employed in mining, almost three-fourths are engaged in the production of bituminous and anthracite coal; one-fifth, in the production of copper, iron, lead, zinc, and other metals; and the remainder in getting out structural and miscellaneous materials from quarries. The meeting of the needs of the coal-mining industry, therefore, is the problem of greatest importance in the point of numbers to be served. Moreover, the dangers in coal mining are greater than in metal mining.

Practically all states have set up educational and technical standards for the position of foreman, boss, superintendent, etc., and every applicant for these positions must have a certificate of competency. The qualifications, too, are becoming increasingly difficult in the field of technical information. In some states, every miner must have a certificate, which is issued by the mine examining board only after an oral examination.

Did these state laws not call for definite and comprehensive plans for the vocational education of men in the mining regions, the conditions

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under which coal and the metals are today brought out of the ground and made ready for the market would of themselves demand systematic practical training for those engaged in such large and increasing numbers in this important industry. Furthermore, since mines and smelteries are often distant from other industries and civic organizations, the responsibility of employers for the welfare of their employees is greater than in most other industries; by the nature of the enterprise, they must assume many of the burdens of public education and social betterment.

Previous to the present war, an increasing number of non-English-speaking men were migrating to mining communities. These were often experienced miners from Europe but were unable to read, write, or speak the English language. Preliminary and fundamental to vocational training, therefore, in many mining communities, is an effective course of instruction in the speaking and reading of English. In most mining districts, this need is understood and is beginning to be met by the public schools or by classes established by the employers. In far fewer instances, however, is it realized that the good of the community, as well as the welfare of the industry, calls also for evening classes, part-time classes, and, in some cases, day schools designed to give all employees sufficient technical knowledge to insure their own safety and that of their fellow-employees, and to provide, for the more ambitious boys and men, opportunities to fit themselves for state examinations, or to advance themselves in other ways.

The usual state school of mines is organized, very properly, as an engineering institution to train leaders in the industry. The course of study requires for entrance the completion of a four-year high-school course. Few of those employed in or about the mines, however, have even entered high school. Statistics for the State of Illinois show that, for every 1000 men, 251 had completed the fifth grade, 680 left school between the sixth and eighth grades, and 69 had completed more than the eighth grade.

A few state schools have been organized as miners' institutes and have offered courses of less than college grade. For example, the Illinois Miners and Mechanics Institute, as a means of offering to ambitious men an opportunity to prepare themselves for passing state examinations for mine inspector, mine manager, mine examiner, and operating engineer, offers: A two-year course of systematic instruction at mining centers; unit courses in mining towns; short course at the university; coöperation with existing organizations, such as the State Mining Board and inspectors, the Mine Rescue Station Commission, officials and locals of the United Mine Workers, the United States Bureau of Mines, high schools and grade schools, libraries in mining towns, first-aid or other local organizations, and the Y. M. C. A.; also, as special activities, organization of local institutes, question and answer department, travel-

ing libraries, laboratory equipment, etc., work for non-English-speaking miners, correspondence courses.

Owing to the failure of the necessary appropriation, this excellent work is suspended; and while other states are doing excellent work in this field, their efforts are sporadic, uncertain, and not coördinate. It would seem, then, that this field invites the coöperation of the Federal Government with the mining states and their local communities to promote methods of vocational education that will make better workmen, a more certain source of supply for positions of responsibility, and greater stability in the personnel of the working forces in the mines. Such coöperation is now possible under the terms of the Vocational Education Act.

The Federal Board is already coöperating with certain states in making surveys of typical industries. During the past year the agent for trade and industrial education in the West Central States held a number of conferences in mining communities with mine superintendents, foremen, and employees in an attempt to discover the kinds of schools and the type of instruction that the Federal Board might approve for vocational schools.

Under the terms of the Federal Act, three kinds of vocational schools may be organized:

The All-day School is intended to give to those who have not yet entered employment instruction that will enable them to receive advanced standing when they enter an industrial occupation. Pupils must be over 14 yr. of age and the school day must cover not less than 6 hr., three of which must be in work on a useful and productive basis. The all-day school organized in cities of over 25,000 presupposes that the pupil has selected a single trade for which he seeks entrance preparation. In cities under 25,000, and it is only with these smaller communities that mining is concerned, the length of the term may be shortened and the course of study varied so that several kinds of shop work may be included.

The Part-time School is organized for not less than 144 hr. per year and for pupils over 14 yr. of age. Instruction may be such as to increase the general civic or vocational intelligence of the pupils. Part-time schools for those who have entered employment may give trade preparatory, trade extension, or general continuation-school instruction.

The Evening School receives only pupils over 16 yr. of age and offers courses that supplement the daily employment of the individual.

These schools may be organized to meet the needs of those employed in and about mines in underground occupations as trappers, muckers, timbermen, roadmen, diggers, etc. The occupations above ground do not differ greatly from corresponding occupations in other forms of industry.

A thorough study of the mining industry is needed to analyze the occupations, specify the operations that the individual must perform, list the technical information the individual must possess as an efficient workman, and prepare a course of study. As a result of the preliminary investigations already made, schools are being organized in the States of Oklahoma, Colorado and New Mexico. Reports from states in other regions indicate that similar schools are under way in Idaho, Nevada, West Virginia, Pennsylvania, etc. During the coming year, it is hoped greatly to extend the work.

The problem before the Federal and State Boards is one that requires the coöperation of the local community, employer and employee, and various mine organizations such as the American Institute of Mining Engineers, the American Federation of Labor, etc. This preliminary statement is made to stimulate discussion among those immediately concerned and to secure their suggestions and their active interest.

## DISCUSSION

J. C. WRIGHT.—The problem of organizing and maintaining a vocational class for those employees who are engaged in the mining industry depends on several most important factors. The first is the sympathetic coöperation of employer and employee; the second is to find the subject matter that must be presented to these men; and the third is to find the instructor who can present that subject matter. This is perhaps the most difficult, for we usually find a man who knows the material that is to be presented but is not a teacher or does not know how to organize his material. The man must know the subject matter from the standpoint of absolute contact with industry in order to be a successful teacher.

I have come into contact with two kinds of schools organized in connection with mining occupations. In one type the instruction leans very largely toward elementary subjects, arithmetic, reading and writing and spelling; it is not of such a character as to help or improve the working man in his occupation in the sense that we are attempting when we speak of vocational education. In the other type of school, an engineer is usually employed as a teacher. He immediately begins to give the things with which he is most familiar and the instruction becomes such as only those who possess a high-school or college education, perhaps, are able to receive. In between these two schools is the school we are trying to promote, a school that will meet the needs of those employed.

MARGUERITE WALKER JORDAN,\* Altoona, Pa.—Vocational education has become the business man's problem; for he is the keeper of that white elephant, labor turnover, and anything that will decrease the size of the

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\*Advisory Council, Industrial Relations.

monster is worthy of serious consideration. This vocational education can do; for it can supply many of those deficiencies in upbringing and environment that make for the discontent and restlessness of our varied classes of unskilled labor: the Anglo-Saxon mountaineer; the Southern negroes who have crossed Mason & Dixon's line by the thousands, "searching satisfaction for our minds;" and the still greater number who have sought across the sea America, "the promised land."

For no one is this question of discontented labor more acute than for the coal operator, especially because the mining industry, by its very nature, offers unattractive home environment. The timber has been largely stripped and many of the streams look like liquid coal. Huge waste heaps, barren hills, grimy red and gray houses help to make a landscape that is monotonous and dreary. Working away from the sunlight is not particularly conducive to mental or moral advancement. Add to this the lonesomeness of the job, time to think and little to think about but grievances, and the fact that the element of danger is always present. Is it any wonder that the miner returns at night, gloomy and irritable, to his unsightly home and half-cooked meal?

Here at once vocational education can remedy much, with its domestic-science courses. The oldest of the world's arts, home-making, is still strangely new to the vast majority of those who assume life's gravest responsibility without the least training. Contrary to the average man's opinion on the subject, women are not born knowing how to sew or cook. We catch influenza and other things, but we do not catch home-making. If the wife of the wage earner knows anything about it, it is because her mother knew; and if that mother was a peasant of Southern Europe, a mountaineer, or a cotton field "nigger," her chances are slim. It is usually almost as much a matter of individual discovery as if she were living on a desert island, but when eggs are 80c. a dozen, Robinson Crusoe stuff comes pretty high. The ordinary domestic-science course of the public school offers little aid to this condition, for familiarity with electric equipment and white marble-top tables gives one no sense of poise when she meets a kitchen equipment consisting solely of a two-eyed coal stove and a frying pan.

Nor are the public schools much more adequate in developing general intelligence. In a recent issue of *Coal Age* I read this: "Let the teachers be instructed by mine officials, to maintain a high standard of order and cleanliness in the school." But what of the fact that 350,000 of our teachers are less than 19 years old; that two-thirds of these have not completed the eighth grade; that a large majority of these are teaching in mining villages, where "them shoes" and "has saw" is good school English? Moreover, we pay the average school teacher less than a day laborer. Our national chewing-gum bill is larger than our educational bill. Can the coal operators look for community improve

ment through such instruction? Yet children at the rate of a million a year are leaving these schools to enter industry, children with practically untrained minds. Naturally, the child and the industry can profit in only a small degree from the instruction of the foreman.

Industry suffers, too, because our schools fail to realize that learning to work and working can become an affair of absorbing interest. Disregard of this work instinct of the child—which is first expressed in play, visualized and acted from the local industries—means abnormal and curtailed development; and this is the beginning of our misfits, unfits, the lame ducks of industry. In contrast to this there is the story of a little girl in one of the mountain schools who had a job of making beds. One day she was not feeling very well and was told that unless she felt much better she need not get up in the morning.

"Oh," she said, "I'm aimin' to be stirrin' soon; hit's Saturday, and I wouldn't trust anybody else to put clean sheets on the beds."

As far as my limited experience in industry is concerned, that spirit of responsibility is the thing mine owners are in greatest need of; and, contrary to former opinions, we have learned that it won't "just grow." This very spirit is a product of vocational education.

There is excellent precedent for vocational education. In 1642, parents and masters were warned "to give their workers training in employments which would be profitable to themselves and to the Commonwealth." England now demands 8 hr. per week of vocational training for all workers under 18, as well as for unskilled employees above that age. This is done on company time, and in this way will England maintain its supply of skilled labor. France, also, has compulsory training in all shops. Our own vocational schools demonstrated their possibilities by training thousands of army mechanics in a short time. Many of America's industries have adopted vocational work, and the Smith-Hughes Act makes it possible in all industries.

Other things being equal, a man of the mines is the ideal teacher, and if he is man enough for the job, the difficulties will iron out. When a person is hired for the work, it ought to be understood that it is not a "two hours a night" job, but a job to which is to be given all the time necessary to do a good piece of work. The school needs an executive head, with time for organization, and one with an understanding of community, individual, and industrial needs. Just as in any business, there must be care used as to its establishment, and such a job cannot be wished on the already over-busy school superintendent.

The vocational school is in reality a business and must take the point of view that if it is going to do business it must adopt aggressive methods of advertising. Of primary importance is the necessity of presenting the work solely from the standpoint of utility. The fact that 50,000 men have enrolled in mining courses of correspondence schools shows the



demand when the value is understood. The time and interest of a miner is limited, and the work must be so organized that he can easily get what he needs. The experienced workers have judgment based on first-hand knowledge; they know by experience what the trade demands of them and what are their deficiencies; they know what things are assets and what things are not. For this reason the courses for experienced and inexperienced workers differ. Classes should be grouped according to age as well as mental capacity. The evening schools in the British Empire were a failure for years largely through the grouping of men and young boys in the same class. There cannot be the ordinary school standards. The entrance qualification is fitness to profit by trade instruction.

The guiding thought to be held throughout the course is the necessity of making the man do the maximum amount of thinking to meet a present, not a future, need; for he will not submit to a preparatory drill. Therefore, the necessity for the short unit course. For instance, to automobile mechanics a course could be given in five lessons in testing and experimenting on lubrication. Such a short course compels direct and intensive training; thus the effort of the pupil is concentrated upon one subject and not dissipated among several. The result is more regular attendance, more intensive work, and a corresponding gain in efficiency. Of course, there can be several short unit courses, arranged in sequence, each giving its own certificate. The knowledge of a definite accomplishment is a greater incentive, and the state examinations are another.

Textbooks are difficult, largely because "they ain't none;" the bulletins furnished by the Bureau of Mines are possibly the best. Due to greatly varied conditions, it would be unwise to map out a standard course. A careful study of the equipment available, both in schools and in private establishments, often reveals unexpected sources of supply and will determine the courses that can be given to advantage.

Labor is the largest cost factor in nearly all kinds of production. We know, for instance, that to maintain a force of 1000 men costs annually from \$100,000 to \$200,000. Therefore, it is obviously even wiser to improve our labor than it is to improve our machinery. It is on this basis that vocational education can be justified as a corporate activity. The Federal Board comes forth with a very liberal offer of aid; yet it does not propose to dictate to the operator what sort of training shall be given to his miners. It specifically states that no sort of training will be forced upon him. He himself will define the training to be given, by simply stating which of his needs he cannot provide for by any system of shop training.

The average employer in the United States has accepted without question the general proposition that he has nothing to contribute to

educational work. He has looked upon schooling, together with the professional schoolman, as something set apart from real life. Now, however, is his opportunity to make a contribution of very real value to education. The methods of vocational education are being developed; the mine owner is asked not to sit back and wait for the vocational school product to come and then find out that it is not what is needed. The producer (the school) and the consumer (the employer) must get together now, in the formative period, and agree on the kind of training needed and how it can best be given; then encourage the boys to take the school training by giving them a chance to make good. It is discouraging for a boy who has given several years to a course of training in a trade school to start on the same level as a boy from the street, who has had no training whatever. Do not sidestep by saying, "If he is any good he will come up to the top." He may, if he has a chance and is not lost in your organization. If the school is any good, encourage those who take the training by recognizing its value when they come to you for employment. If it is not what it ought to be, take an active interest in it and help make it what it should be. It will pay in satisfaction and in dollars and cents, for it will give the student a distinct advantage in the market. Success is conditioned upon the development of a degree of efficiency equal to, if not surpassing, the degree attained by competitors. Do the mine owners of America want to utilize this fund for improving the efficiency of their own employees, or shall the Federal appropriation be turned back into the Treasury as not wanted?

It has been said that there is hardly a kind of agony on the modern battlefield that has not its counterpart somewhere in our economic struggle. Can we not face this struggle in the same high spirit with which we faced the struggle of arms? While it is true that the war released the lowest and grossest traits of humanity, it also tapped wellsprings of spiritual strength, of idealism, of self-sacrifice, of service and devotion at which cynics and pessimists and criminal minds of the world have stood aghast. Are we to lose all this because the war is over? The value of coöperation has been demonstrated as never before; the twentieth century has made us into a brotherhood; and the very industrial relations that offer exasperating chances for misunderstanding, discord and collision, offer also noble opportunities for that greatest of undertakings, the upbuilding of humanity.

E. A. HOLBROOK,\* Washington, D. C. (written discussion†).—My purpose in discussing this paper is to emphasize to the members of this Institute the fundamental and far-reaching changes in the present ideas of training miners, which this nation-wide plan of vocational education soon will put into effect. Any mining company, knowingly or unknow-

\*Acting Chief Mining Engineer, U. S. Bureau of Mines. †Received Feb. 17, 1919.

ingly, is under considerable and constant expense in breaking in new men. Today every man going underground for the first time must learn from haphazard contact and chance, little by little, the duties of his vocation and how best to guard himself against the peculiar dangers and conditions underground. The army has shown what it is possible to do in the way of concentrated training of a group of men in any chosen vocation. When vocational schools are established in every important mining community, it will mean a uniformly high standard of knowledge of his duties by every man who calls himself a miner.

In mining communities in the United States, efforts in vocational educational lines have heretofore often been isolated, irregular, and confined to a few of the largest mining communities, or carried into effect by a few of the larger mining companies. Often, the man employed as teacher has been chosen for his qualifications as a school man, and with minimum regard for his ability and practice in the vocation he teaches. In some cases there has been an effort to make a partly trained engineer of the student, rather than a first-class craftsman at his trade. In other cases, universities and states have, or are working on, first-class standards of vocational education. Considering the great extent of the mining industry, and the inadequate sums of money hitherto allotted for the purpose of vocational education, the efforts have not reached the great body of miners in every state, which is now possible under Federal supervision and aid.

Consider the problem from a purely economic viewpoint. Our mines are every day getting deeper and larger, and machinery is constantly replacing hand labor. These improvements in mechanical and electrical appliances have increased the complexity of the trades belonging to the mining industry and necessitated a greater knowledge in the workers. On the other hand, this introduction of machinery means that we are no longer wholly dependent for manual labor on the skilled American miner and the immigrant from Britain and the north of Europe, who in many cases come from families who have been miners for generations. Of the Scotch, English, and Irish miners in this country, 92 per cent. were miners in the old country. The use of machinery has enabled men to gain employment in mines as loaders for mining machines and in other underground occupations, whose only qualification is that of a laborer. Thus, the last 20 years has seen a tremendous influx of south Europeans into our mining industry. These men have had no experience in mining, generally having been agricultural laborers; for example, only 3.6 per cent. of the Lithuanian and 7.3 per cent. of the Russian miners in this country had previous mining experience.<sup>1</sup> The employment of these men underground must ever be a menace.

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<sup>1</sup>Statistics from the Study of the Immigration Commission in 1910; an exhaustive report, which includes the immigrant in the mining industry.

Mining is a hazardous occupation. Mr. Wright states that the dangers in coal mining are greater than in metal mining. Although this is the generally accepted idea, a study of the accident statistics of the Bureau of Mines reveals that for the last 7 years, 1911 to 1917 inclusive, of about 700,000 men engaged in coal mining 3.38 per thousand employed were killed each year; in the metal mines during the same period, of about 175,000 men engaged, 3.79 per thousand per year were killed. Statistics compiled from the report of the mine inspector of Pennsylvania for the years 1915 and 1916 combined show that the miner of so-called south-European races was, in proportion to the number engaged, nearly twice as liable to meet with fatal accident as the native and foreign miner of the English-speaking races. If education in his vocation were to put this man on a level of skill equal to his more fortunate brother, there would be a great saving to the mining companies of many millions of dollars spent in compensation insurance, in the cost of hiring and firing men, and in increased efficiency of the workmen. In addition to this financial saving, there would be a tremendous gain to the country on account of the good-will, contentment and loyalty gained by Americanization of the foreign miner.

The work of the various mining companies in having elaborate safety organizations and in conducting safety propaganda, which appeal to the eye and ear of the miner, and the work of the Bureau of Mines in promoting safety measures, must be considered partial expedients looking to the time when fundamental education in the trades shall make the man of the new generation more skillful than the present, both in his work and in handling himself underground. It must be, then, to the interest of every mining company to actively help this vocational work through the introductory stages.

CHARLES F. WILLIS,\* Bisbee, Ariz. (written discussion†).—There are few mine officials who will not say that there is great need of vocational schools in mining communities, but the absence of success of so many of these ventures makes them wary about establishing such schools. They have neither the time nor the facilities to study the systems and to analyze the factors that have made certain schools a success and others a failure.

In the first place, it is difficult to secure the proper instructors. Educators cannot make the appeal to the workmen, because of their lack of experience, and experienced men who are capable of the appeal are not usually educators, knowing nothing about the presentation of the subject. Then, too, there is ordinarily no objective in a mine school. The

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† Received Apr. 15, 1919.

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workman is not reached by a school that makes him merely a more valuable man to his employer, unless he can participate in the gain. An objective, as promotion or financial gain, would appeal to the man and would make for regular attendance.

Usually vocational schools are run like all others—a number of subjects at a time, continued from one day to another. This is impractical for the workman, whose mind cannot work like the mind of a school boy. The school boy is spending most of his time in school work, while the man is doing it as a side issue, and he cannot be expected to pick up to-night's lesson where last night's left off. The workman is accustomed to the idea of completing a job and doing one thing at a time; his mind is trained that way, and for this reason a school that works on the principal that each lesson is a complete job, with no reference to the previous or following lesson, will go a long way toward removing one of the things that has caused many failures.

The usual vocational schools in mining communities are either very elementary, or are run by the engineering staff and, consequently, are too advanced. There is no plan between. It is believed that there is a possibility of a halfway type, but this is a subject that is large enough for a paper by itself and is entirely too broad to be presented as a discussion.

Another important feature in the discussion of the establishment of vocational schools is the number of sessions per week. The teacher believes that they should be often and continuous; it is usually forgotten that the man has other obligations, social and recreational, and that he cannot live for work and school alone. He is going back to study after years of doing something else evenings, and he cannot suddenly change his habits and eliminate all other activities, which, through years of use, have become a part of his life. If anything is to be dropped, it will be the newest thing, the school. The man who has developed none of these social and recreational habits is not the type of man that can be reached by any school.

The lecture method of instruction expects the ordinary workman to do something that many men with years of training cannot do: grasp the spoken words and retain them. Text-books used in mining vocational work are written for college use and are generally beyond the comprehension of the workman. Moreover, no text-book, or even a dozen text-books, covers the whole subject, and the expense is a serious consideration.

This discussion is not intended to decry the movement for vocational education, as the writer believes it is perfectly possible to work out a vocational-school plan that will consider the above difficulties, but the question should be carefully studied in order that progress may be made by eliminating the reasons for former failures.

J. C. WRIGHT (author's reply to discussion\*).—It is true that so-called vocational schools for miners have failed to function to a very large degree. Mr. Willis has mentioned a number of the factors to be charged with the responsibility for this failure. These factors may be summarized as:

1. A failure on the part of those responsible for the instruction to analyze mining occupations as the first step in determining the character of the subject matter to be given.

2. A lack of teachers possessing the necessary qualifications.

3. The failure on the part of those in charge of these schools to relate the instruction to mining occupations and to organize the subject matter on a short unit basis.

4. The failure to secure qualified teachers.

5. School organizations that have attempted to make the vocational mining schools parallel either academic schools or mining engineering schools, the one resulting in instruction of a purely academic character and the other in instruction too far advanced and too technical to be assimilated by the great body of those employed in mining occupations.

6. A lack of text-book and reference material written in a simple style, suitable for men who possess less than an elementary school education.

The provisions of the Vocational Education Act, as well as the policies of the Federal Board, have in many ways provided the necessary safeguards to prevent frequent repetition of the above undesirable factors. Some of the safeguards around which the Act was framed and the policies and activities of the Federal Board affecting the above factors are:

1. A study and investigation of the field of vocational education in mining communities has been under way for several months. This investigation is being made by A. C. Callen, as special agent for the Federal Board. It is expected as a result of this survey to have published and ready for distribution by the next fiscal year the following bulletins: A general bulletin on organization and administration, and an outline of short unit courses in which classes may be organized; a bulletin on mine gases; a bulletin on mine ventilation; a bulletin on mine timbering; and a bulletin on safety lamps.

The subject matter of the bulletins is being prepared by individuals who have been selected because of their intimate knowledge and experience in mining occupations from the standpoint of a skilled employee. In addition, a study is being made of the occupations above ground for which training may be given.

2. Under the terms of the Vocational Education Act, each state has a state Board for Vocational Education charged with the responsibility

of disbursing Federal funds in the training of teachers for vocational schools or classes, and in the promotion of evening, part-time, and all-day vocational schools. Under this Act, the qualifications of a teacher in an evening, part-time, or all-day class who is to give technical or shop instruction in mining require that he must be a man of practical experience gained through actual participation in mining occupations. These qualifications are such that an individual selected as a teacher of classes in mine ventilation, mine gases, and mine timbering must be a workman with more than average intelligence and experience in these subjects as they relate to mining. A mining engineer without practical experience would not be satisfactory.

3. All of the foregoing bulletins are being prepared so that instruction may be given on a short unit basis. The individual may attend the class for a definite number of evenings and complete a course in shot firing, in mine timbering, in mine ventilation, or on safety lamps.

4. Each State Board is required to set up in its annual plan the qualifications of the teachers who will be employed in the schools or classes for which Federal aid is asked. In all states, these qualifications are based on the experience of the teacher in industry, on his technical knowledge of the industry, and on his ability to instruct. Recognizing that workmen drawn from the occupation are without skill as teachers, each state has submitted plans for training teachers for these schools. This training may be given in evening instruction, by correspondence, by itinerant teacher training, or by resident instruction at institutions.

5. The Vocational Education Act requires that all instruction in approved schools must be of less than college grade. It is therefore impossible to approve work of the grade usually given in engineering institutions. The Act also provides that all instruction must be given in separate and unmixed classes.

The above safeguards have been thrown about the Federal Vocational Education Act in order to make the instruction effective and in order to direct it toward the seven or eight million men and women employed in trade and industrial pursuits.

## Engineering Work of the National Research Council

BY HENRY M. HOWE\*

(Milwaukee Meeting, October, 1918)

1. *The purpose* of the National Research Council as organized for war purposes is twofold, to stimulate those outside its own personnel to conduct researches of importance for winning the war and to carry on such researches through its personnel to a limited extent. How this is done is explained in Section 10. "Research" is used here in a very broad sense, including, for instance, inventing and developing mechanical and physical devices.

The need of this work arises from the inevitable concentration of most of the governmental war agencies on the production and transportation of war materials of the types now adopted, as distinguished from devising new kinds of instrumentalities, such as armor, guns, aircraft, and apparatus for detecting, locating, observing, signaling, transporting, and many other military purposes. But it is important that we should devise new agencies. The war came near being won by an invention, the submarine, and its course has been affected greatly by two other inventions, tanks and aircraft. In spite of our numerical superiority victory may well be snatched from us by an invention, if we allow Germany to outstrip us in inventing. Hence the importance of the Council's work in mobilizing and organizing the services of the patriotic civilian experts throughout the country, so as to bring their powers to bear on important war problems and on inventing war devices, as an adjunct to the development work of the regular governmental bodies.

2. *Status of the Council.*—Though President Wilson's request caused the Council to be created in 1916 by the National Academy of Sciences under its congressional charter; though his executive order of May 11, 1918, directs the heads of "Governmental departments immediately concerned" to "continue to co-operate with it in every way that may be required;" and though the Government has contributed largely to its financial support; it is not a department of the Government but an independent research body, aiming to evolve the necessary mechanism for the novel work of systematic stimulation and guidance of research. Thus the Government, which instigated the creation of the Council, recognizes and collaborates with it, and in part supports it.

The Council acts moreover as the Department of Science and Research of the Council of National Defense.

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\* Chairman of the Division of Engineering of the Council.



3. *The war organization* consists of nine divisions, as follows:

- I. Division of General Relations.
- II. Military Division.
- III. Division of Engineering.
- IV. Division of Physics, Mathematics, Astronomy, and Geophysics.
- V. Division of Chemistry and Chemical Technology.
- VI. Division of Geology and Geography.
- VII. Division of Medicine and Related Sciences.
- VIII. Division of Agriculture, Botany, Forestry, Zoology, and Fisheries.
- IX. Research Information Service.

4. *The Organization of the Division of Engineering.*—It is made up of four sections as follows:

- 1. Prime Movers. L. S. Marks, *Chairman*.
- 2. Mechanical Engineering. W. J. Lester, *Chairman*.  
Committee:  
Fatigue of Metals. H. F. Moore, *Chairman*.
- 3. Metallurgy. Bradley Stoughton, *Chairman*.  
Committees:  
Helmets and Body Armor. Major Bashford Dean, *Chairman*.  
Ferro-Alloys. J. E. Johnson, Jr., *Chairman*.  
Steel Ingots. Lt. Col. W. P. Barba, *Chairman*.  
Pyrometer. George K. Burgess, *Chairman*.  
Improvement of Metals by Treatment at a Blue Heat. Zay Jeffries, *Chairman*.
- 4. Electrical Engineering. C. A. Adams, *Chairman*.  
Committee jointly under Metallurgy and Electric Engineering; Electric Welding of Ships. H. M. Hobart, *Chairman*.

We expect to organize additional committees in the near future.

These sections are directed by an Executive Committee consisting of the Chairmen of the Division and of the four Sections.

In addition there is an Advisory Committee consisting of:

Van H. Manning	Officially representing the	U. S. Bureau of Mines.
Pope Yeatman		War Industries Board.
D. W. Brunton		Naval Consulting Board.
Ambrose Swasey		Engineering Foundation.
George S. Webster		American Society of Civil Engineers.
Philip N. Moore		American Institute of Mining Engineers.
John R. Freeman		American Society of Mechanical Engineers.
C. A. Adams		American Institute of Electrical Engineers.
A. A. Stevenson		American Society for Testing Materials.
W. B. Price		American Institute of Metals.
Edward P. Hyde		Illuminating Engineering Society.
W. H. Burr	}	Members at large.
Gano Dunn		
George C. Stone		
W. R. Walker		
F. M. Waring		
L. B. Stillwell		

The Advisory Committee meets at intervals to direct the general policy of the division. All the other committees are actively engaged on their several problems, though one of them has been delayed by illness and other complications.

5. *The sources of our problems* are very varied. Some come from various governmental bodies, some from the industries and the engineering societies, some from inventors and the general public, and some from our own staff.

Problems and projects the solution and development of which promise to be useful either for war or peace, for industry or for science, are our grist. By sending them to us this Institute and the engineering profession in general will enable us to broaden our service to them, and indeed to mankind. So, too, we welcome offers of coöperation from professional experts, whether through work done in their libraries and laboratories, through sending us important data, or otherwise.

6. *Researches by Experts Outside of our Personnel.*—Let me illustrate these by means of certain researches which the Section of Metallurgy has in hand.

The winning of the war is retarded appreciably by the delay caused by the rejections in making certain important kinds of forgings, such as shells, crankshafts, cannon, etc. These rejections are due chiefly to faulty procedure in making and forging the steel ingots themselves. The faultiness exists because the rapid increase in the demand for such ingots has led many into this manufacture without sufficient experience. The problem thus is "How can we bring the procedure of the relatively inexperienced up to that of the best makers?" We are trying to solve it by means of our Steel Ingot Committee, composed of men who are recognized by all as among the most capable experts on this subject in the world, under the chairmanship of Lieutenant-Colonel W. P. Barba, himself one of the highest authorities. It contains representatives of the Ordnance Department, the Bureaus of Standards and of Mines, Bethlehem, Midvale, the United States Steel Corporation, the Illinois Steel Company, the Crucible Steel Company of America, and the Standard Steel Company. We may strengthen this committee still farther by adding to it French and British experts of like eminence.

We expect this committee to prepare a set of detailed directions constituting "Recommended Practice." Appearing over names of such weight it should immediately become standard. Its existence should not only instruct the manufacturers needing instruction, thus reducing the personal equation to a minimum, but also strengthen the hands both of those drawing up contracts and of the inspectors who enforce them. Moreover it should do this at one motion for all branches of the Government.

7. *Pyrometry.*—One of the points on which definite instruction should

be given for this very matter of ingot making is the temperature of the molten metal in the open-hearth and electric steel furnaces. Because no pyrometer for determining this temperature exists, a necessary step in our evolution is to devise one. This, though in part a question of pyrometry, is also in large part one of refractory materials.

To solve this problem a committee has been formed under the chairmanship of Dr. George K. Burgess, of the Bureau of Standards, and with representatives of Leeds & Northrup for pyrometry; of the Norton Company, the Joseph Dixon Crucible Company, and the Harbison-Walker Refractories Company for refractory materials; and of the Midvale Steel and Ordnance Company and the Taylor Wharton Iron and Steel Company for steel making. These two steel making companies have placed their open-hearth and electric furnaces at the disposal of the committee for this work.

8. Our other committees are working in like manner on problems of immediate importance for war-winning. Thus the Committee on the Fatigue of Metals, under Professor H. F. Moore, of the University of Illinois, is directing its study toward the endurance of aircraft crankshafts and of the electric welds in welded ships. That on Ferro-alloys, under J. E. Johnson, Jr., is studying the saving of manganese along three distinct lines, that of bringing the work of the least successful smelters up to that of the most skillful ones, that of replacing part of the manganese used in steel making with other deoxidizing agents, and that of lowering the manganese requirements of existing engineering specifications.

This work incidentally calls for the determination of many melting points of the combinations of oxides resulting from the substitution of other deoxidizing agents for manganese. A large number of research laboratories at many educational institutions and industrial works are collaborating in this difficult work.

This general study is carried on jointly by this committee and the United States Bureaus of Mines and of Standards and the Geophysical Laboratory.

The study of the treatment of metals at a blue heat is in the hands of a committee headed by Dr. Zay Jeffries, Director of the Research Department of the Aluminum Castings Company, which has contributed so much to the development of our aircraft. This difficult study promises results of great value.

9. *Electric Welding.*—The Committee on the Electric Welding of Ships, under the chairmanship of Mr. H. M. Hobart of the General Electric Company, is making an exhaustive study of the technical and especially the metallurgical problems which arise in substituting electric welding for the customary riveting of steel ships. It is a division of the Electric Welding Committee of the Emergency Fleet Corporation. Its investigations are carried on at many different works and laboratories,

notably those of the General Electric Company and the Bureau of Standards.

10. *Researches by our own personnel* are illustrated by the Section of Mechanical Engineering, under Chairman Lester. It has long had its own engineers and draftsmen, and it now has in addition the laboratories and machine shop of the Carnegie Institute of Pittsburgh. By means of these the rather nebulous but attractive inventions offered us are developed in coöperation with the Division of Physical Sciences, into definite concrete form, in which the military authorities may judge quickly as to their present and future utility. In some cases we complete the apparatus fully enough for actual service tests.

Among the inventions which this section is now developing are these:

1. A special gun for use on aircraft.
2. A special drive connected with it.
3. Control for aircraft.
4. Aircraft propellers.
5. Aircraft fuel.
6. Tanks of various types.
7. Mechanism for the control of trucks.
8. Special type of tractors.
9. Special telescopes.
10. Special balloons.
11. Parachutes.
12. Special aircraft motors.

Military considerations prevent indicating more closely the nature of these inventions.

The stages in which these devices now are vary all the way from preliminary design to nearly complete readiness for production.

In addition we have before us many other promising proposals which we cannot develop for lack of funds.

11. *Government Support.*—In some cases, after we have shown clearly enough that a given invention is of real promise, we receive an appropriation from some military or other governmental body to enable us to carry the development still farther. In other cases the research is taken over by some governmental body. Thus the work of the Committee on Helmets and Body Armor was taken over by the Ordnance Department many months ago, and is still carried on by that organization and the Council jointly, its chairman, Dr. Bashford Dean, being commissioned as a major. This accords with our general aim of originating and stimulating research. We shall accomplish more in the end if we leave to others the completion of the researches which we have led them to undertake. Our work is not so much to investigate as to cause investigation, cheerfully forfeiting the credit due the investigator proper. So, too, a device may be materially changed after it leaves our hands.

### **Mental Factors in Industrial Organization\***

BY THOMAS T. READ, E. M., PH. D., NEW YORK, N. Y.

(New York Meeting, February, 1919)

READJUSTMENT of the industrial world to a peace basis after more than 4 years of war will involve many fundamental and far-reaching changes that cannot as yet be clearly foreseen or definitely provided for. Such problems may be subdivided, in the vocabulary of war, into those involving material and those centering about personnel. Of the two, the latter group is the more difficult and obscure, as the factors involved are less clearly understood and the methods to be followed are not yet tested and standardized. When it is said that the market is weak, stocks large, and demand light, every business man understands clearly what is meant and what course he should adopt to adjust himself, so far as possible, to such conditions. But when it is said that the rapid spread of Bolshevism in Europe is a danger to industrial organization in this country, it is difficult to have any assured sense that all that the statement may mean is clearly understood or that the means to be followed to meet such a situation are definitely known. Since Bolshevism is primarily a mental phenomenon and since the rupture it brings about in industrial organization should be prevented, if possible, it is my purpose to restrict this discussion of the activities of the Committee on Industrial Organization to some of its mental aspects, as the chairmen of the sub-committees, from their special knowledge of their own fields, can best set forth recent progress in the lines with which they are especially concerned.

The first requisites of success in industrial organization, as in the biological organism of which it is the social counterpart, are unity of purpose and coördinated activity. Typical good and bad examples of this are the relative parts that the United States and Russia have played in the great war. These two factors are primarily mental, and it is not difficult to view the mental aspects of all problems of industrial organization as the most important ones. Unless we can attain unity of purpose and coördinated activity in industry, the provision of comfortable homes at reasonable rent, the assurance of steady work at good wages, the elimination of industrial accidents, and provision against want in old age or in case of disability will have brought us no nearer to our real goal.

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\* Report of Chairman of Institute's Committee on Industrial Organization.

An industrial organization is made up of three groups: those who furnish the capital and take the risk of the enterprise, but frequently have little active part in it after it is firmly established; those who represent the first class in the active direction of the enterprise; and those who give their energies for an assured wage, without assuming any of the risk. Some of the reasons why these three elements find it difficult to achieve unity of purpose and perfectly coördinated activity are indicated below, in the hope that their statement in this brief form will draw forth helpful discussion.

Activity of any kind, up to the fatigue point, is agreeable to any human being, if it has an adequate motive and offers an opportunity for self-expression. Under such circumstances much effort may be put forth and great discomforts endured with equanimity. A good example of this is duck-hunting; very few people care to spend more than a few days in this form of activity, for the motive declines in relative importance and the discomforts relatively increase. Under most forms of industrial organization, the workman has little motive for labor, beyond the wage offered; his opportunity for self-expression comes in the character of the work done. The growing complexity of industrial processes due to research, and the immigration into the eastern United States of large numbers of workmen of relatively low intelligence, has lead to the development of the functional, or so-called scientific, system of management. This method was developed by F. W. Taylor as the best way of supervision of men of inferior grades of intelligence. Its great drawback when applied to workmen of higher intelligence is that it almost completely robs them of the few opportunities for self-expression in their work that remain under the modern system of assigning only one operation out of many to an individual. The management still finds its motive and self-expression in putting on the market a product that can be sold at a good profit, but the workman is too remote from this to feel any satisfying part in it, although the industrial value of an established brand or trademark must not be overlooked, since the workman finds some satisfaction in laboring to turn out a product of recognized merit. Safety work has accomplished some good along this line by what is known in psychology as deflection; the workman does a standard amount of work without any accident and finds his opportunity for self-expression in the latter feature of it. Company baseball teams, bands, first-aid teams, and similar activities that enable the individual employee to express his personality are of value. But the net result of the present tendency in industry is for the workman to see in his work only the unpleasant necessity of earning a living and to find his mental satisfaction in other forms of activity. This is putting the cart before the horse; it is just as though the army went to defeat the enemy only as an unpleasant necessity and found compensa-

tion for its sacrifices in something else. The various profit-sharing schemes that have been devised are only a makeshift and do not introduce the workman as a partner into the enterprise in the sense that his motive becomes identified with that of the organization. The fundamental fact remains that the workman is not in a position to take much risk, and how he can be made a partner in industrial enterprise without sharing its risk is an unsolved problem.

The wonderful possibilities of an adequate motive and properly coördinated activities has been indelibly impressed on every business man who visited the training camps of our National Army and saw the results accomplished in a few months' time with what must have been slightly below average human material, since many of the most effective men secured industrial exemption. No one who has seen it can forget the tense eagerness of both officers and men. An industrial organization that could inject anything like the same spirit into its personnel would be, like our army, invincible. Coördination of activity depends on leadership; identification of motive in industrial organization is impossible unless the principle is admitted that the opinions of the laboring members of the organization are entitled to a respectful hearing and consideration on their merits.

Underlying these general considerations are the mental characteristics of the individual workman. Perhaps the most significant of these for our present purposes is the necessity to the individual for rationalizing his acts, or the assigning to them of a motive that is satisfactory to himself. A man, for example, strikes another who has insulted him, not because he is angry, but because honor requires it; or he refrains from striking him, not because the other is too large to strike with impunity, but because it would be too undignified to do so. In this way the most unworthy acts can be put on high moral grounds. Sabotage is a typical example of this in industry. The mental reaction in a man who can make only a poor living by hard work and is confronted with the easy success of others more fortunate is too apt to be one of jealousy and the end product the adoption of a "what's the use of trying" attitude. We all talk of equality of opportunity and have too much tendency to overlook the fact that it is largely beyond the control of ourselves or anyone else. Two soldiers of equal merit may be assigned to the infantry. One gets into battle, captures an enemy gun, escapes unhurt, is decorated, returns in triumph, and is elected mayor of his home town. The other contracts pneumonia and dies in a field hospital. Up to the limit of human control both had equal opportunity. It is admitted that both were of equal merit, yet the reaction many of their colleagues will exhibit is sympathy in the one case and a feeling that "I could have done it better if I had had the chance" in the other. When with this latter feeling is coupled a tendency to rationalize a wrong course of action, the individual

becomes very badly adjusted to the circumstances under which he must live. A rough definition of a Bolshevik would be a person who is dissatisfied with things as they are and is able to rationalize a course of action that experience indicates will make them worse instead of better.

The next point to be here considered is that efficiency is in large part a question of mental adaptation. Dr. Yerkes is to present to our membership an account of the methods that have been developed under the auspices of the War Department to determine the relative intelligence of men as an indication of their fitness to become officers. The trade tests developed by the same committee are of even greater interest in industrial organization. Capital has become too cheap and human effort has become too dear to admit much longer of the time-honored method of trial and error as a means of fitting the job to the man. The man who is not fitted to his job is constantly having the ground cut from beneath his feet by discouragement and discontent, two arch enemies of efficiency. The greater part of progress along this line has yet to be achieved, for we have made only a beginning as yet.

There are many other normal mental factors that might profitably be discussed but they must be left for consideration at another time in order to pass on to the second half of this subject, the relation that mental abnormalities bear to industrial organization. It is now definitely known that some people exhibit throughout their lives mental abnormalities and others exhibit them at times. The paranoids form one group. If these are of the depressed type, they are surly and suspicious, believe themselves to be unjustly treated, and fail to appreciate any kindness shown them. The Kaiser has been described as an example of the exalted type, persons who believe they are benefiting others while making infinite trouble for them. Another definite group comprises the emotionally unstable, who show a tendency to brood and be unhappy, but are modest and self-effacing. They are always ready to undertake anything new, but tire of it before it is completed. They are marked by the violence of their reactions to slight provocation. It is definitely known that these types are present in industrial organization, but neither their numbers nor their effect on the normal psychology of the organization has yet been ascertained. In addition to these permanently abnormal people are those who are temporarily deranged. People who cannot stand worry, overwork, or other unfavorable conditions give way mentally and one-fifth of those admitted to insane asylums each year are of this type. Most of them later get well. There may be hundreds of thousands of cases of mental disturbance of this kind that do not reach the point of requiring admittance to the asylum, just as a man may have a severe cold but not go to the hospital. It seems reasonable to suppose that the paranoids may have a large effect in breaking down unity of purpose and coördination of activity in a normal body of workmen and that the emo-



tionally unstable may play a large part in inciting the violent outbreaks that sometimes attend industrial disputes. Since mental abnormalities of this kind are relatively more common among people of marked ability than among the mediocre, it is hardly necessary to add that mentally abnormal persons may be found in the management as well as in the working force. It is the hope of this committee that it will be able to do some pioneer work in ascertaining the prevalence of the mentally abnormal in industry, determine the actual effects, what means should be taken to minimize the bad effects and to utilize to the fullest possible extent whatever possibilities there may be for good.

### Discussion on Housing

(New York Meeting, February, 1919)

D. EPPELSHEIMER,\* Middletown, Ohio.—The housing of employees has so many and so varied aspects that in order to reach even an approximately correct solution it is necessary to have in mind a few fundamental principles; but it is not possible as yet to write these housing principles in the clear and correct form that is possible with many of our natural laws. Manifestly, the problem depends first on the kind of people it is necessary to house. What is suitable for the Southern unattached negro is not suitable for the married skilled American. The influx of colored labor has affected quite a number of what were satisfactorily housed communities.

The second important factor is the relative proportion of the number of employees to the total employable population of the community in which the industry is located. The employer of a hundred men in a large city has little responsibility in the matter of housing his employees. If he employs 75 per cent. of the employable population in a community of 27,000, as is the case of the company with which the writer is connected, it means a large responsibility. In the establishment of an entirely new plant in a more or less isolated place, the responsibility becomes greater.

As engineers, we design and write the specifications for the machinery to accomplish a definite purpose, and proceed to house it, provide the necessary buildings with their accessories in the way of cranes, or heating systems, or sprinkling systems, as the case may be. There is no reason why the operating force should not be built up in very much the same way: design the organization, write its specifications so that the employment department can secure the proper men, and then house that organization with just as much thought and care as is given the plant. In the plant, the various machines will work along certain definite lines and accomplish certain results; but even if we start with the proper organization it takes considerable time to get that organization working together. One of the most important elements to this end is the proper housing of the organization. A man usually spends more time at home than at work. Proper surroundings and a comfortable, convenient house of good appearance exercise an influence that cannot be denied, and an employee situated in such surroundings is far more inclined to follow the policies of his employer and take an active part in furthering them than one dissatisfied with his home and surroundings, which feeling

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\* American Rolling Mill Co.

is accentuated, if not exaggerated, by his family who are among these surroundings all the time.

*Housing Methods of American Rolling Mill Co.*—It might be well to explain briefly the experience of the American Rolling Mill Co. as it may be helpful to others in solving similar problems. Its housing efforts are divided into three classes: (1) Housing laborers and semi-skilled men, providing quarters and meals; (2) housing foreigners, by providing houses that are owned by the company and rented to individuals; (3) through a realty company, developing vacant lands in various sections of the city and building houses that are sold to people having different incomes.

For the first class of employees, there are six bunk houses, 22 ft. by 150 ft., which accommodate 800 colored men. These are equipped with double-deck beds and each man has his own locker. A full crew of cooks is kept; since some men work 8 hr., others 10 hr., and others 12 hr., meals must be served in the dining room at 5:30, 6:30, and 8:00 A.M., 12:00 M., and at 3:00, 6:00, and from 11:00 P.M. to 12:30 A.M. This room accommodates 264 men at one time. For those that cannot go out for dinner, packed buckets are provided. This provides the bare necessities of living, but the company believes that this is but the beginning so it has provided a hospital, a commissary where the ordinary wants, such as gloves, overalls, tobacco, etc., can be obtained, a recreation hall, and a school teacher and books for those who want to learn the three R's.

For the unmarried skilled employee there are in the community opportunities for boarding; but as these did not prove sufficient at one time the Bachelors Club was provided. This house has 20 rooms and accommodates 42 persons, provides meals, and has all the accommodations of a college dormitory. It is under the supervision of a man who, in a sense, is a head master.

For the foreigner, there have been purchased or built 58 houses, having a total of 300 rooms. Several are 8- to 12-room houses and practically one-half are 5-room houses. As a place for recreation, the Foreigners Club has been provided.

This plan takes care of the housing of the employees who receive the laborers' rate; but what about the skilled employee, the foreman, the superintendent, and other employees in an organization of 4800 men? This is where the relation between the size of the community and the size of the industry plays such an important part, and when the number of employees is 60 to 75 per cent. of the employable population, the responsibility of the company becomes very great.

In addition to the unattached men there are a number of colored men drawn North by the higher rates of wages, who left their families in the South. The employer has a fine opportunity to give these a chance to get out of their thriftless, slovenly way of living, by providing a really

respectable place of living, and giving them a chance to become useful citizens. Our colored citizen is not like the thrifty foreigner. He has to be helped a little; but when he is helped he will live up to his surroundings; so the American Rolling Mill Co. has provided four-room cottages for him. These can be purchased by paying 10 per cent. down and 1 per cent. a month. The company's experience has been that the men will make the original and monthly payments as soon as they see the framework going up, and three to four months before they can move in. Some of the purchasers of last year are getting familiar with business ways and pay by check; and in many other ways are living up to their opportunities. They have built their own church, and the company has presented to the board of education, for the colored settlement, a school which has a large assembly hall that can be used for community purposes. For the men who are in the semi-skilled class, there have been built other houses.

During the war the question of housing was brought to public attention in a more forcible manner than would have been possible in times of peace. The progressive industrial manager has felt its influence, but has in many cases been unable to impress its importance upon those responsible for the company's policies, and secure sufficient support, especially in the matter of finances that permit the housing matter to be handled in a thorough and comprehensive manner. Happily, in the case of the American Rolling Mill Co., those responsible for the company's policies have led it along the lines that have just been explained. But the company is just beginning. The problems unsolved are just as important and perhaps just a little more involved, but we have had considerable experience; and as we begin to see the fruits of our experiments, we will take great pleasure in trying to solve those still before us.

As a real recreation, aside from the industrial daily work, a recreation that will call for all your knowledge of human nature, your artistic temperament, your utilitarian sense—for houses must be of the utmost utility—and your ideas of construction as well as economy of construction, permit me to commend to your earnest attention the question of housing.

LAWRENCE VEILLER,\* New York, N. Y.—I spoke to you last year of the rather critical situation that confronted the country at that time in the great shortage of housing. I suppose the reason advanced by the mining interests for not building good houses is the old idea that the mine is only going to last a short time, and that it would not pay to build any substantial houses; but as a matter of fact these "temporary" buildings stand for many years.

Most of the mining towns of the United States existing today have existed for many years and are likely to go on for many years to come; even if they are only to be there for five or ten years it is advantageous to

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\* Director, National Housing Association.

pay attention to the housing of the employees. I am not making this appeal to you on the basis of social uplift but rather on the basis of your own selfish interest. If there were no other reason for building good houses for the workers, the dictates of self-interest ought to be controlling.

After the period of readjustment and during the period of financial and industrial depression, which we may have, and in the boom time coming after that period, when we are really working at top-notch speed, we are going to suffer more in this country from lack of labor supply than from any other factor. This is obvious when you realize that we are not likely to have much immigration and we are likely to have considerable emigration in the near future; if that is so, there is naturally going to be a shortage of labor supply.

The employer of labor, the manufacturer and the miner, will not, when that time comes, be able to hold labor merely by higher wages or shorter hours but will do so by making life more attractive to the laboring man. The simplest, easiest and quickest way to do this is to begin with the home; for, as Mr. Eppelsheimer has said, a man spends more time in the home than he does in the shop. How stupid it is for the employer to say, "These men are no good. We shorten their hours and they ought to have more pep." He never stops to think of the conditions under which the men are living. How can the men have any pep when they must live as so many of them do? Many of them sleep in a house where there are several bunks; the air is foul and there is the discomfort of sleeping with a lot of other men and breathing in the odors that come from others. Is it any wonder that in the morning they haven't any "pep"? They do this day in and day out and finally they can't stand it any longer and quit. The employer asks why they are leaving and they don't know; they are just tired and move on to another town hoping to find something better.

The minute you start to do something for the working man your associates will say, "Look out for paternalism. The working man is very much opposed to paternalism." I have never seen a case where you gave the working man low rent and good living conditions that he thought it was paternalism. What these men object to is a lot of fussy people trying to manage the way they shall live. You needn't be afraid at all that good housing will breed paternalism. If you are afraid of paternalism, there is an awfully easy remedy, and that is democracy. Let the men who live in the houses have a say in the management of them. Have a House Committee, made up of the men, to pass upon all complaints. It is a perfectly simple proposition. In conclusion I want to say that there isn't anything the mining engineers of the country can do to aid the development of the mining properties that will pay for the effort expended anything like as much as will the proper housing of the employees.

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